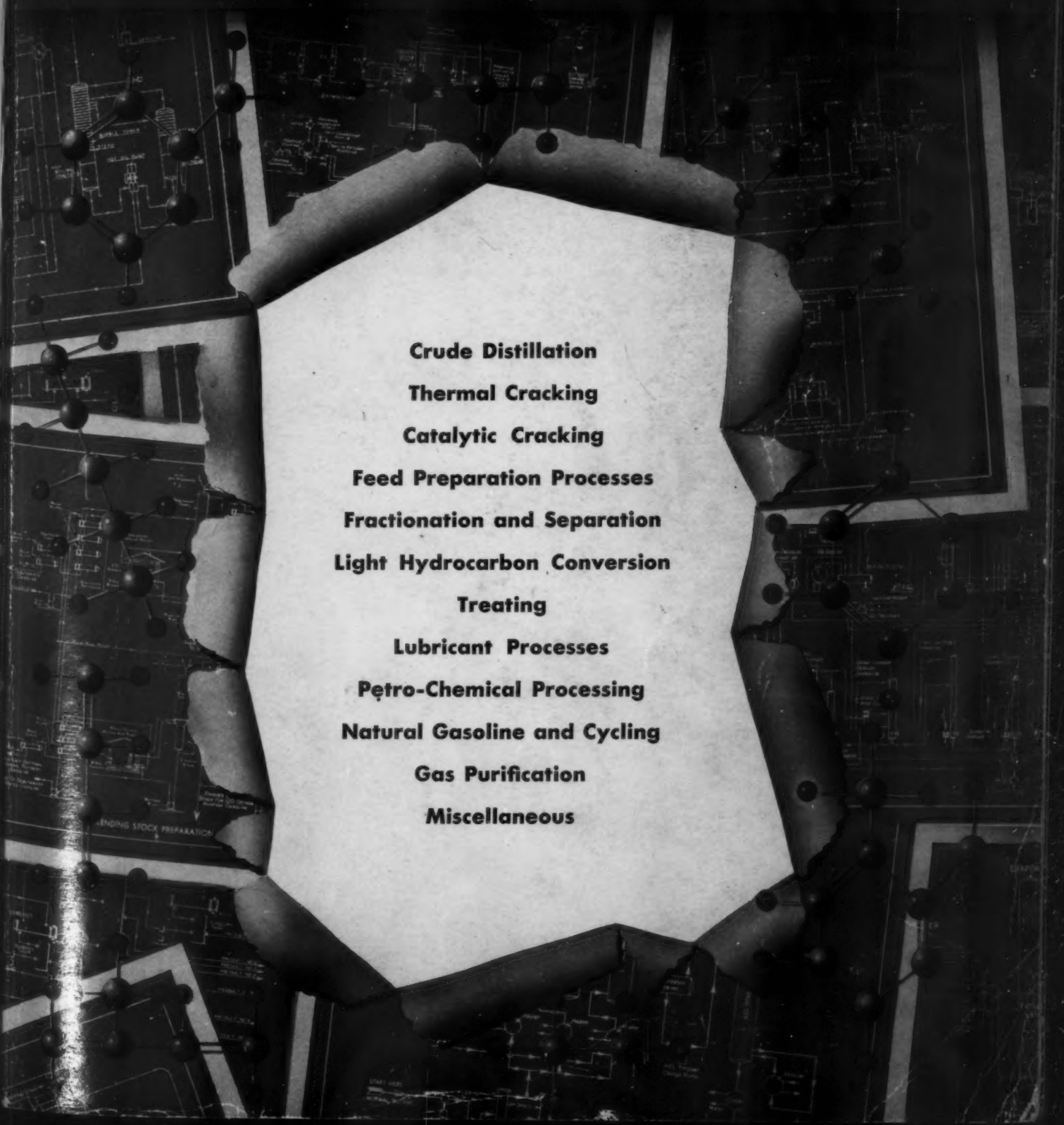
PETROLEUM REFINER

1947 PROCESS HANDBOOK



Crude Distillation

Thermal Cracking

Catalytic Cracking

Feed Preparation Processes

Fractionation and Separation

Light Hydrocarbon Conversion

Treating

Lubricant Processes

Petro-Chemical Processing

Natural Gasoline and Cycling

Gas Purification

Miscellaneous

Neale

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Process design of the plant pictured was developed by the Research and Development Department of The Atlantic Refining Company. All other phases of the project including the field piping systems were developed and completed by HUDSON in collaboration with the Engineering and Construction Department of The Atlantic Refining Company.

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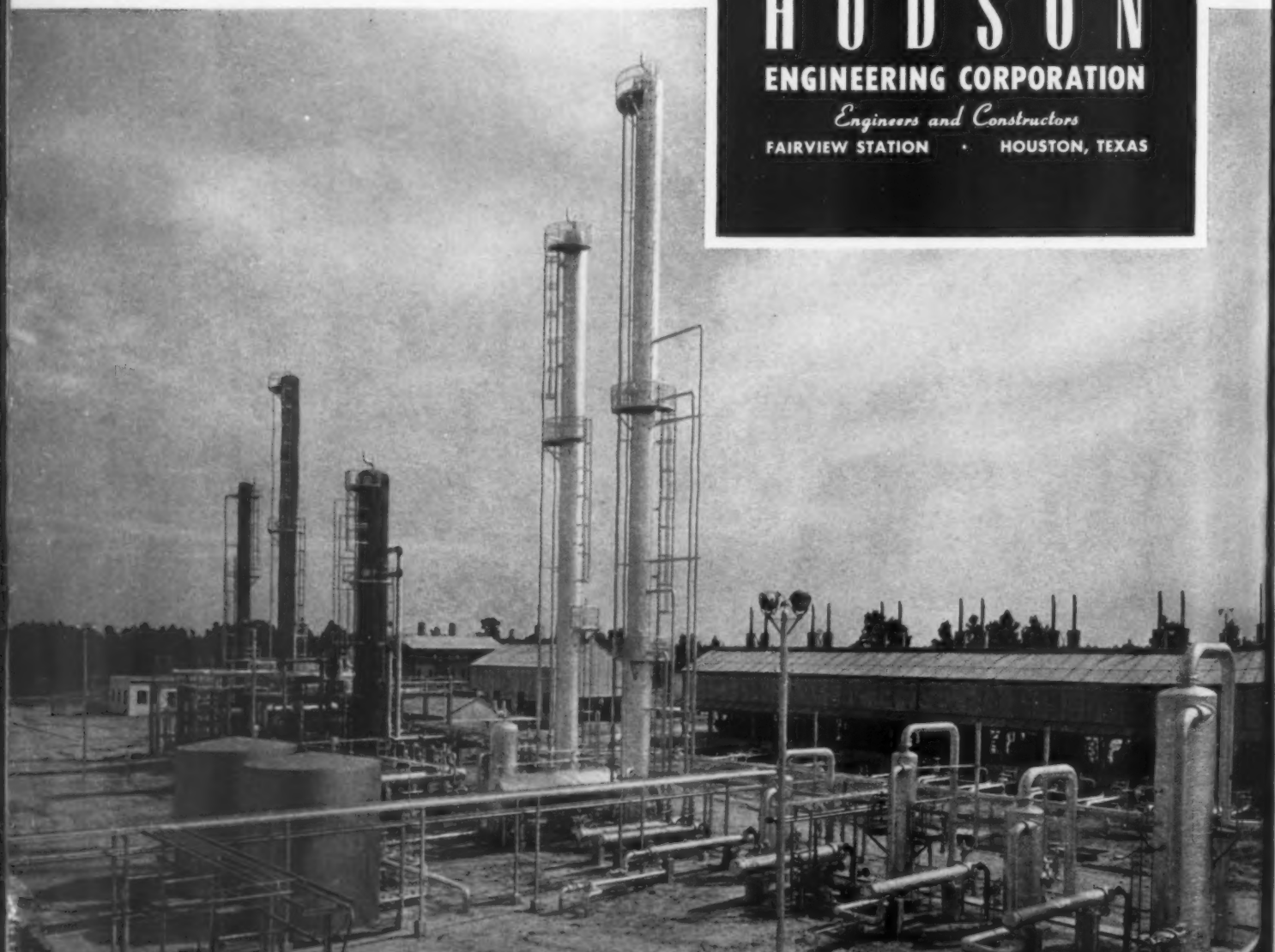

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PETROLEUM REFINER

SPECIALIZED FOR THE OIL REFINING INDUSTRY, INCLUDING PETRO-CHEMICAL ENGINEERING, PETROLEUM SYNTHETICS AND NATURAL GASOLINE

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VOLUME 26, NO. 4 APRIL, 1947

1947 Process Handbook Section

Processes ..... **94 to 295**

Including:

Distillation
Thermal Cracking
Catalytic Cracking
Fractionation and Stabilization
Alkylation
Isomerization
Polymerization
Lubricants
Treating
Petro-Chemicals
Cycling
Natural Gasoline
Gas Treating

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This PROCESS HANDBOOK Section of PETROLEUM REFINER was compiled as a part of Volume 26, 1947, for distribution with the April issue, Number 4 of that volume. Distribution is coincidental with subscriptions as of April.

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Petroleum Refiner is indexed by Industrial Arts Index.

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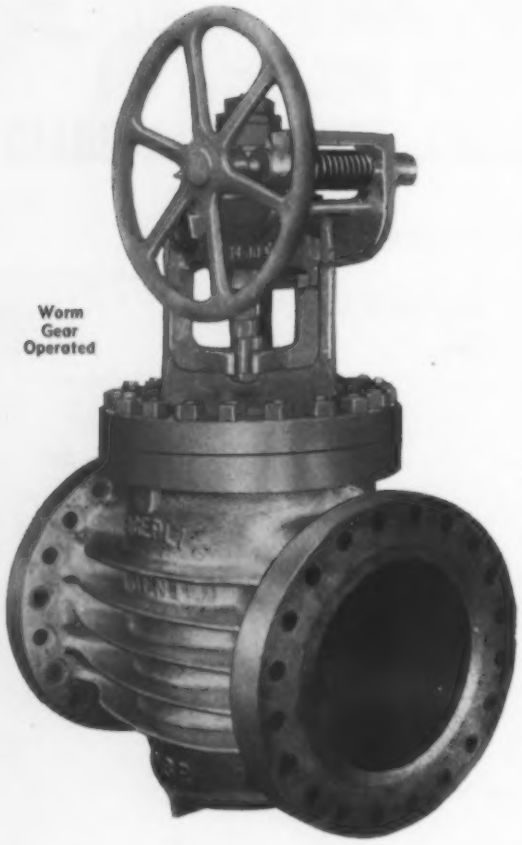

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ATTAPULGUS CLAY COMPANY,
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Phthalic Anhydride from Petroleum
Propane Deasphalting Process
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Dow Styrene Process

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Filtrol Fractionation Process

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Precise Naphtha Fractionation
Thermal Reforming Process
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Vapor Recovery System

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Girbotol Process
Hygirtol Process

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Houdry Fixed Bed Catalytic Cracking Process
Houdry Process for Treating Gasoline
Houdry Dehydrogenation Process
Thermofor Catalytic Cracking process

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Propane Recovery Process

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Fluid Catalytic Cracking Process
Hydroforming Process
Phenol Extraction Process
Propane Deasphalting Process
Propane Decarbonizing Process
Propane Dewaxing Process
Sulphuric Acid Alkylation Process
Vapor Phase Butane Isomerization

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Ammonia from Natural Gas

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Naphtha Polyform Processes
Special Naphtha Rerun Process
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Three-Stage Crude Distillation Process
Two-Stage Crude Distillation Process
Lubricating Oil Rerun Unit

MAX B. MILLER & COMPANY, INC.,
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Perco Copper Sweetening Processes
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Sharples Bari-Sol Process
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Shell Phosphate Process
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Butadiene Process
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Hydrogenation Process

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Indiana Isomate Reaction Process
Indiana Isopentane Process

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Furfural Refining Process
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Gray Clay Treating Process
Hydrocol Process
Osterstrom Clay Treating Process
Solvent Dewaxing Process

TRETOLITE COMPANY,
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Tretolite Desalting Process

TULSA BOILER AND MACHINERY COMPANY,
P. O. Box 1978, Tulsa, Oklahoma
Tulsa Type Absorption Process

UNION OIL COMPANY OF CALIFORNIA,
Union Oil Building, Los Angeles, California.
Azeotropic Fractionation Process
Cobalt Molybdate Desulfurization Process
Hypersorption Process

UNIVERSAL OIL PRODUCTS COMPANY,
310 South Michigan Avenue, Chicago 4, Illinois.
Catalytic Cracking Process
Catalytic Dehydrogenation Process
Catalytic Polymerization Process
High Temperature Clay Desulfurization Process
Paraffin Alkylation Processes
Thermal Cracking Process
Thermal Reforming Process
Unisol Mercaptan Removal Process
UOP Fluid Catalytic Cracking Process

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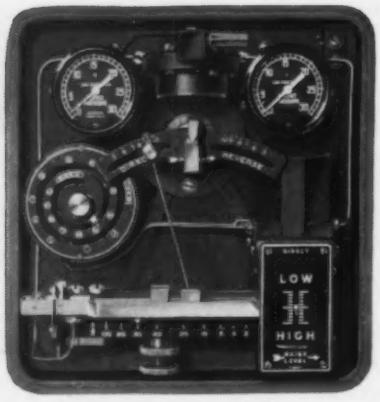

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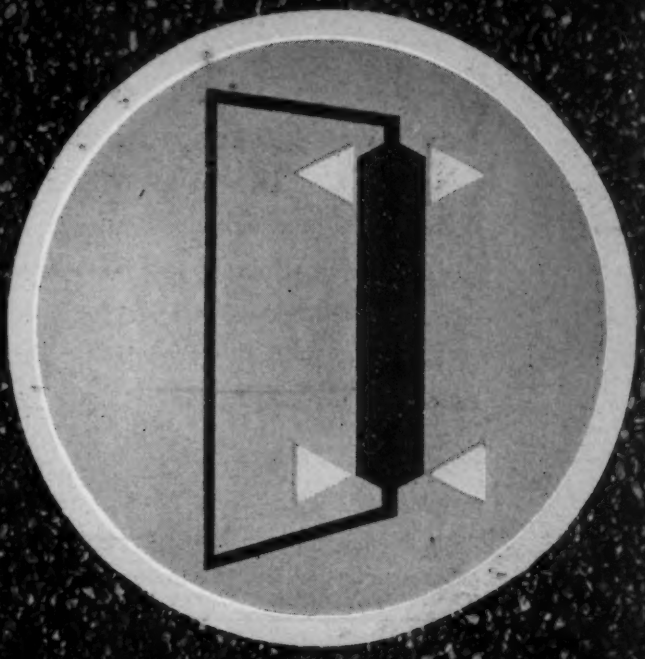


hypersorption

for

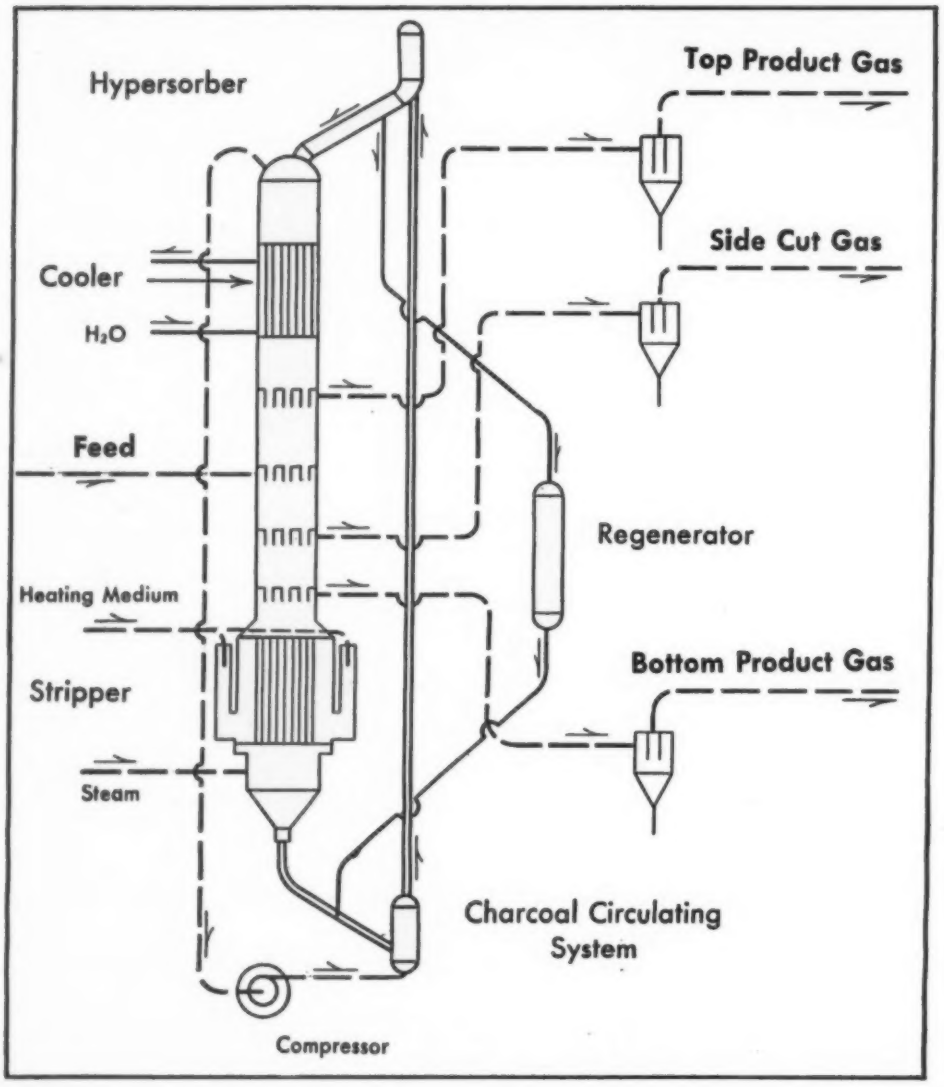
separation of gases and light vapors

FOSTER WHEELER

hypersorption process

The Hypersorption process was developed by Union Oil Company of California for the separation of hydrogen and light hydrocarbons which are not readily handled in conventional separating processes. The process depends on the ability of activated carbon to adsorb heavier hydrocarbons or larger molecules to a greater degree than light hydrocarbons or smaller molecules.

Referring to the flow diagram, it will be noted that the Hypersorption unit has three major sections: cooling section at the top, adsorption and rectifying section in the middle and steam stripping section at the bottom. An elevator or gas lift is employed to convey the granular charcoal from the bottom of the unit to the top in a closed cycle of circulation.



The feed gas enters through a distribution plate and passes upward countercurrent to a moving bed of activated charcoal. In its passage through the bed, the heavier compounds are adsorbed and retained on the charcoal. The top product gas disengages from the charcoal below the cooler and passes off through a cyclone. The charcoal passes downward below the feed point to the rectifying section where it contacts a reflux of heavy

This Hypersorption column is shown under construction at our Carteret, New Jersey works. It will be erected at the Midland, Michigan plant of the Dow Chemical Company. • The photographic view is from the top down, showing (a) the cooling section, (b) the charge and product manifolds and (c) the heating section. The heat is supplied by a Dowtherm vapor heating system.

constituents liberated in the bottom of the stripper which serves to liberate lighter constituents. A concentration of these intermediate constituents in the vapor phase results, and they can be disengaged as a sidecut product gas, at a point midway between the feed and bottom product gas disengaging section. As the charcoal flows past the bottom product gas-disengaging section into the steaming section, it meets an upward flow of steam which liberates the adsorbed heavier components, and these flow upward either to return as reflux or pass out as bottom product gas from the Hypersorber.

The charcoal flows from the steaming section into the stripping section wherein it passes down through tubes which are heated by a circulating heating medium and through which steam is passed countercurrent to the charcoal. The hottest point in the stripper is at the bottom of this section and substantially all residual hydrocarbons are removed during the passage of the charcoal through this zone.

A charcoal regeneration unit is provided to act in parallel with the Hypersorber for the purpose of carrying out high temperature steam treatment of the charcoal to maintain its activity.

The charcoal is transported to the top of the tower by means of an elevator or gas lift system and is dropped into the reserve charcoal hopper at the top of the unit. The hot charcoal passes down from the hopper into the cooling section.

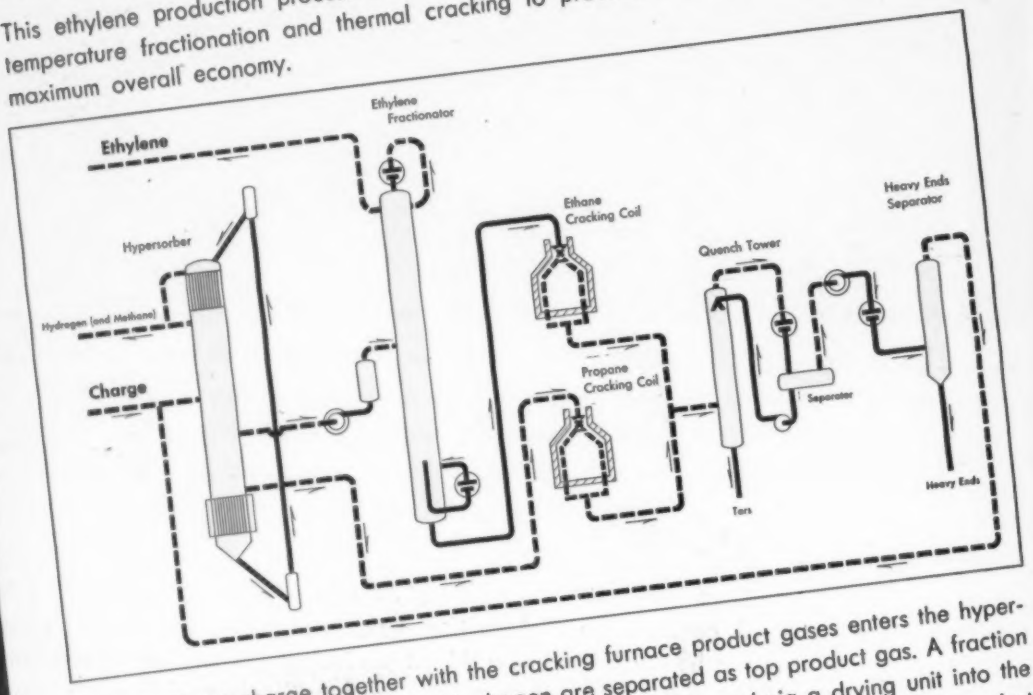
Typical separations which have been accomplished in pilot plant operation:

Mixed-gas Charge	*Product*
Methane-hydrogen-ethylene	Ethylene
Methane-ethane-propane	Propane
Methane-hydrogen-acetylene	Acetylene
Flue Gas	Carbon-Dioxide
Methane-hydrogen-ethane	Hydrogen

hypersorption
in ethylene manufacture

Ethylene, which has attained great significance in recent years as a basic ingredient in the synthesis of plastic, resins, rubbers and similar materials, is a by-product of cracking and is found in refinery fuel gas mixed with hydrogen, methane, ethane and propane.

This ethylene production process combines a sidecut hypersorption operation with low temperature fractionation and thermal cracking to produce a high purity product with maximum overall economy.



The mixed gas charge together with the cracking furnace product gases enters the hypersorption unit in which methane and hydrogen are separated as top product gas. A fraction rich in ethylene and ethane is removed as sidecut and charged via a drying unit into the ethylene fractionator from which commercially pure ethylene is produced. The bottom product of this fractionator is charged to the ethane cracking furnace while the bottom product of the hypersorber is charged to the propane cracking furnace. The outlet gas streams from the two cracking furnaces enter a quench tower where tars are removed. The remaining gases are compressed, cooled and charged to the hypersorber unit via a heavy-ends separator for removal of entrained hydrocarbons.

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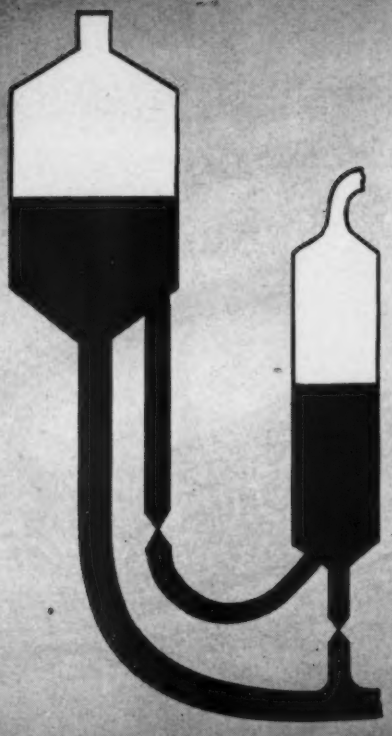
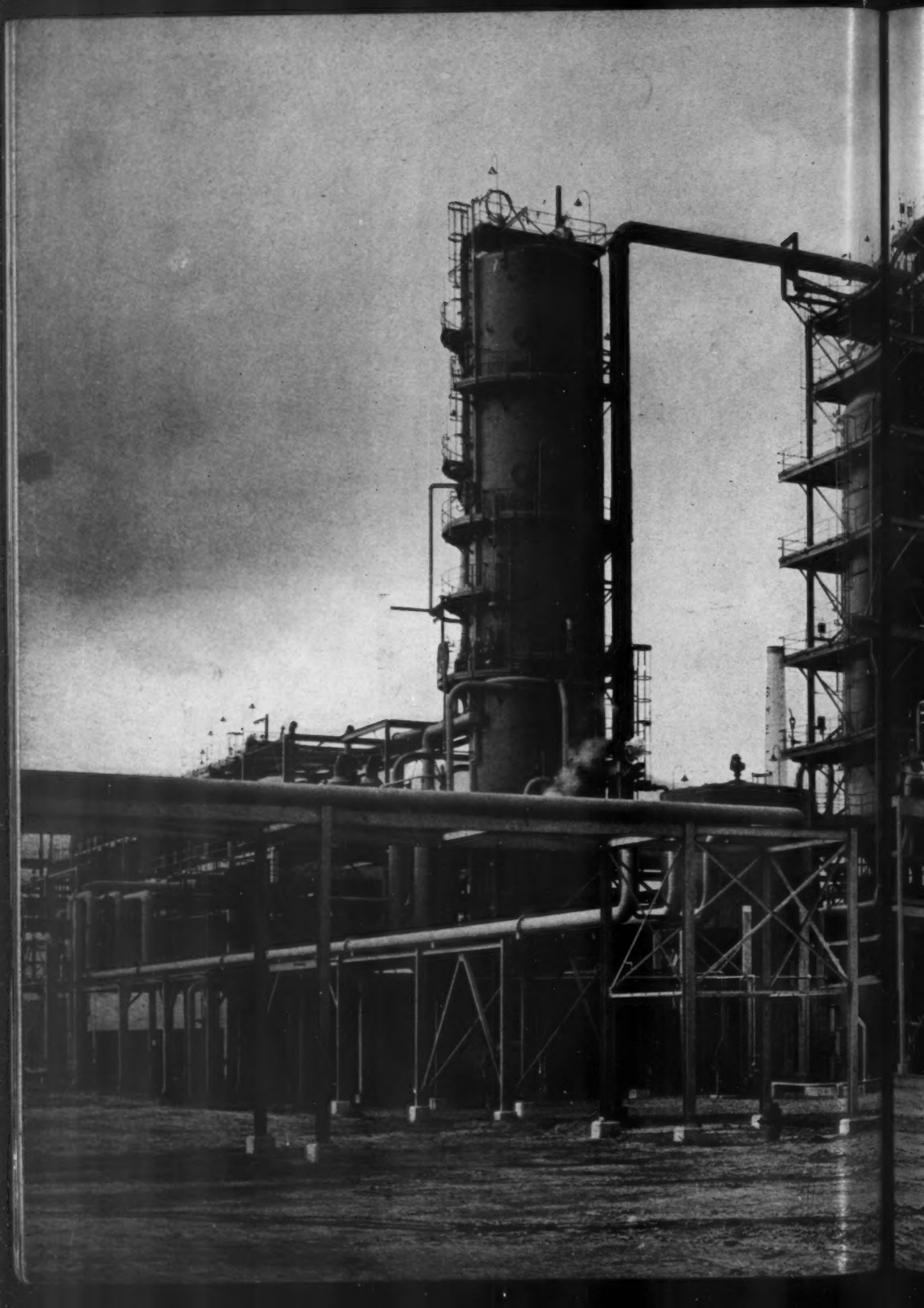
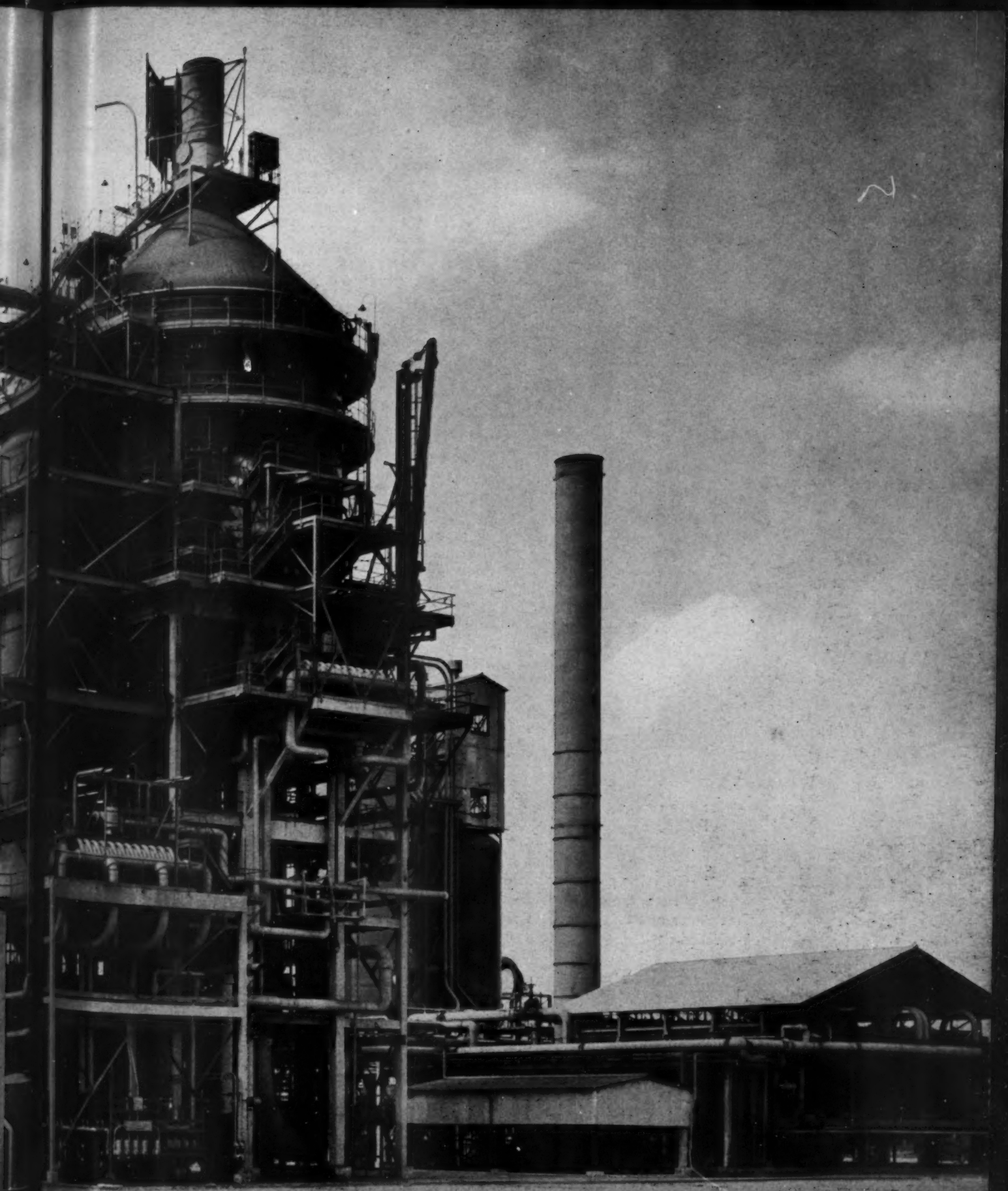
fluid catalytic cracking



. . . the refiner's answer to high research octane

FOSTER WHEELER

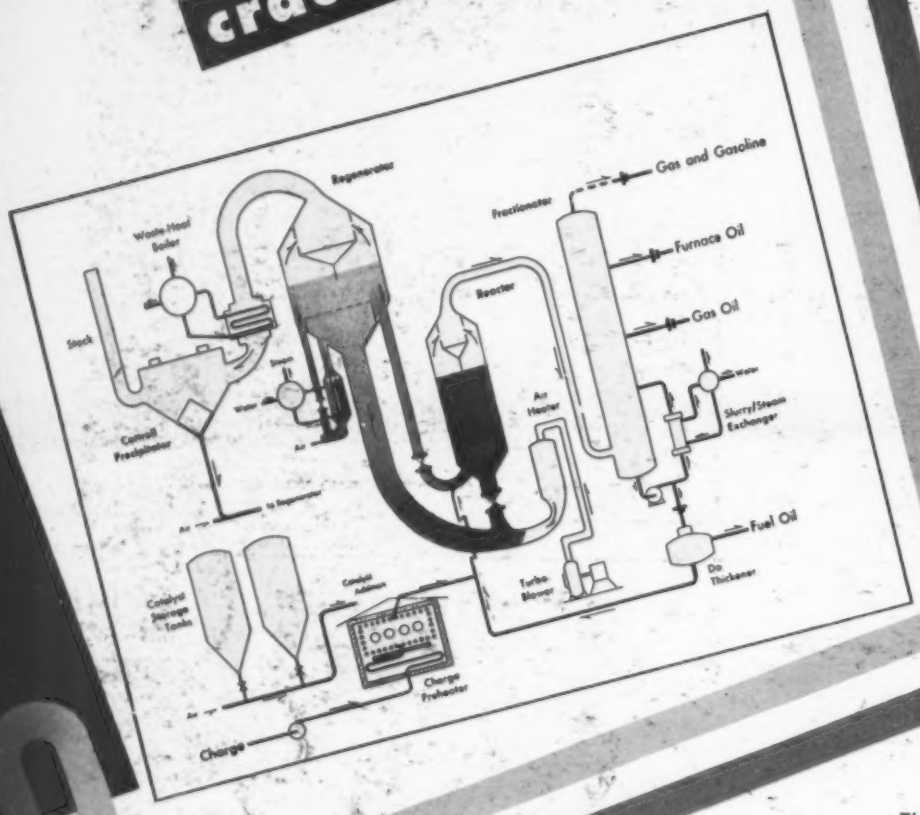




"Fluid" process flexibility is clearly demonstrated in the following comparison of design and operating conditions of this Shell Oil Company unit at Houston, Texas: designed for maximum aviation base and alkylation stocks, at 17,000 bbls. per day charge and 70% conversion, the unit was recently operated for maximum motor gasoline production at 25,000 bbls. per day charge and 50% conversion. Such flexibility provides an effective octane control of the finished motor fuel.

(Designed and constructed by Foster Wheeler in collaboration with the personnel of the engineering and research departments of the Shell Oil Company, Inc.)

fluid catalytic cracking



The flow diagram shows a direct-fired "Downdraft" hea
raising the temperature of the charge to some predet
mined level between 400° F. and 700° F. *(It is not nec
sary to vaporize the charge in Fluid Catalytic crackin*
The preheated oil leaving the furnace, meets and mi
with the regenerated catalyst as the latter flows
of the standpipe from the regenerator. The catalys
a temperature of approximately 1100° F. complet
vaporizes the charging stock, and the reaction
completed in the reactor. The reaction products p
thru cyclone separators, a device in which a sw
ing effect is given to the vapor, throwing out
entrained catalyst and permitting the vapor
pass out of the reaction system almost complet
free of catalyst. The vapor then enters a fracti
ator where the desired fractional separat
takes place. Excess heat is recovered in
waste-heat boiler from which substan
quantities of steam are produced by in
change with the hot residue (or slurry)
culated from the bottom of the fractiona
thru the waste-heat boiler and return
to the column. Any catalyst which
entrained with the vapor entering
fractionator is concentrated in the
due. It is further concentrated in
Dorr thickener and returned to
reactor system.

In the reactor, practically all of
catalyst settles out after react
with the petroleum vapors. The c
alyst, now coated with carb
flows out of the reactor and me
the air for regeneration and is c
veyed by that air into the regenera
where the burning off of the carbon is complet
The regenerated catalyst passes out of the regenera
in the standpipe thru which it flows to meet the incoming cha
ing oil. The temperature in the regenerator is controlled by circulati
the regenerated catalyst thru a waste-heat boiler. As in the reactor, cyclo
separators in the top of the regenerator remove nearly all of the entrained catal
The products of combustion pass thru a flue gas waste-heat boiler where substantial quanti
of steam are generated, thus cooling the gases. Catalyst still entrained, is finally removed in a Cott
Precipitator and returned to the regenerator system. Catalyst may be added or removed by utilizing sim
air-injection devices.

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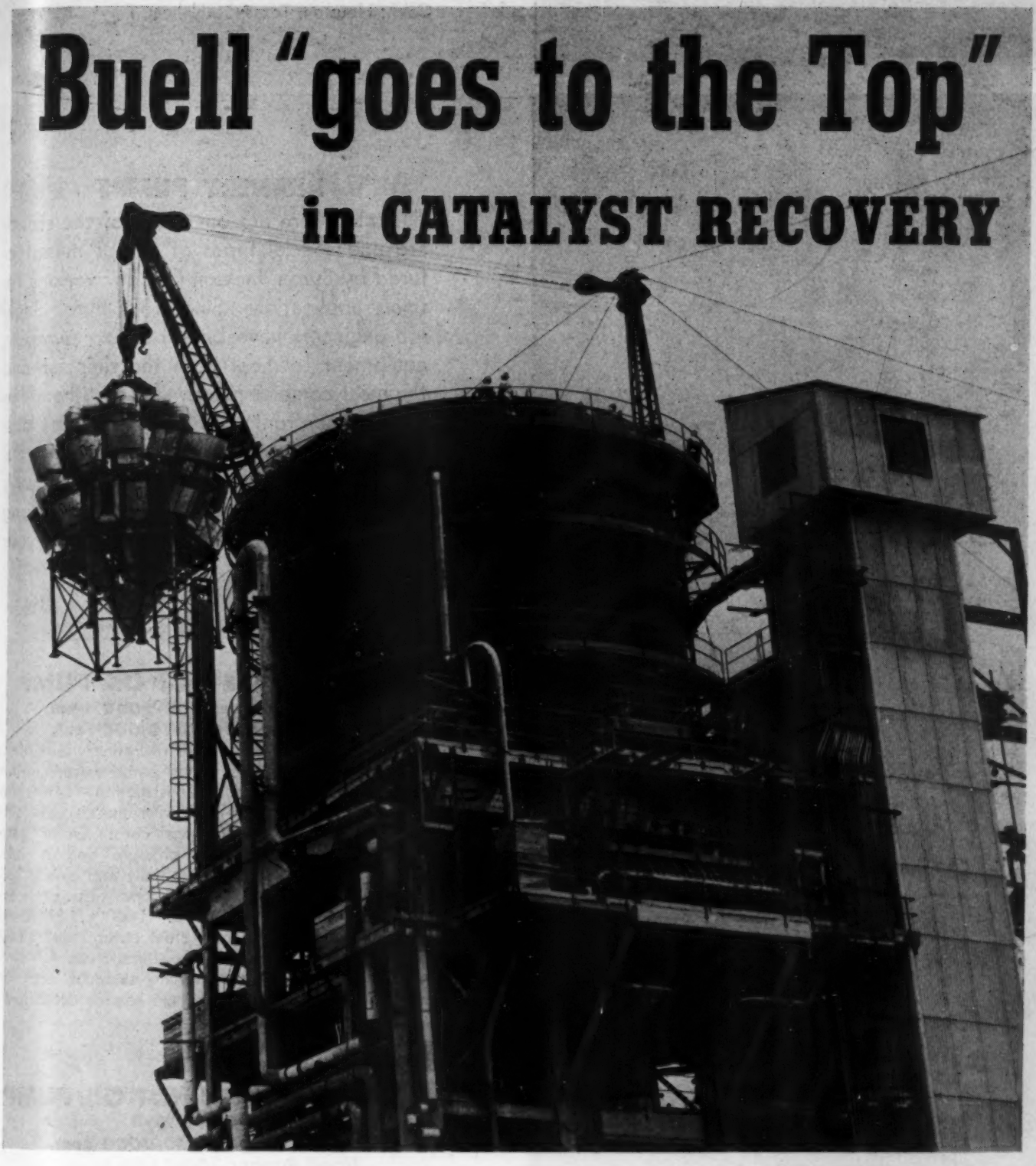

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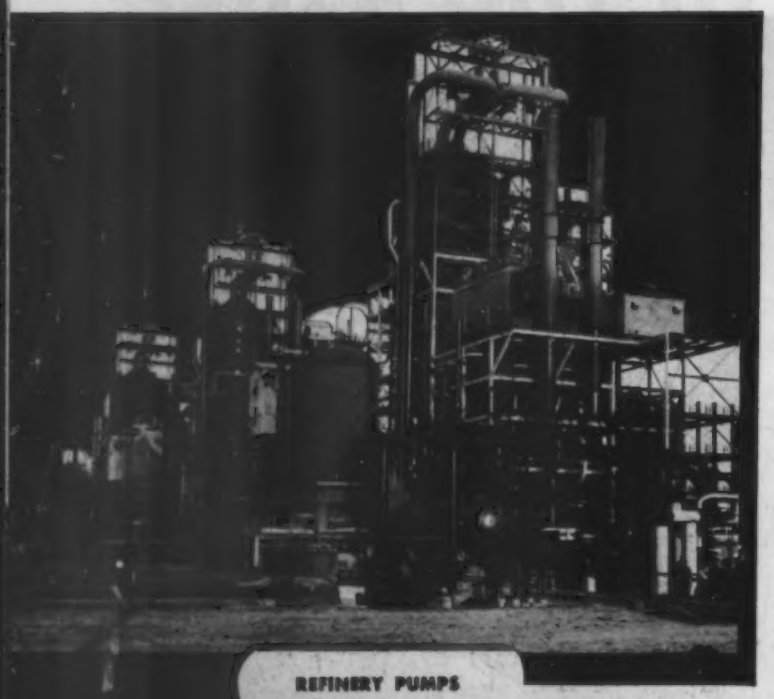
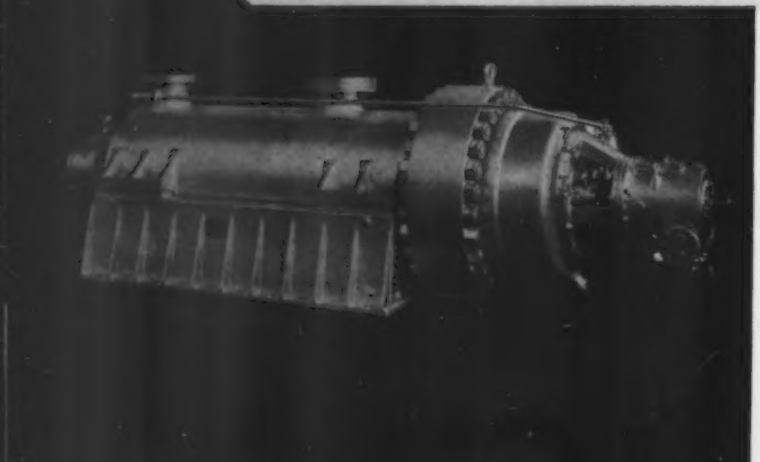


REFINERY PUMPS

REFINERY PUMPS

Illustrated here are but a few of the standard and special types of pumps manufactured by Byron Jackson Co., for various refinery applications. Since 1872 Byron Jackson engineers have been building pumping equipment to meet both the simplest and the most complex needs. If a fluid handling problem confronts you that the pumps illustrated will not solve, before you write your pump specifications call in Byron Jackson's seasoned hydraulic engineers . . . their long experience and knowledge is at your service.



DOUBLE CASE HOT OIL PUMP

DOUBLE CASE HOT OIL PUMP

Capacity Range: 150-2000 gpm.
Head Range: 1200-10,000 feet.

Adaptable for a wide range of refinery pumping, unit is primarily engineered to pump extremely hot and/or volatile liquids. Construction features a simple cylindrical outer case with one circular joint and longitudinally split inner case. Outer case alone withstands bursting pressure and is subjected to tension stresses. Radially and axially balanced, requires no balancing drums, discs or floating seals. Handles liquids to 1000° F. Standard materials: Forged or cast steel outer case; nickel cast iron or chrome alloy steel inner case. Chrome alloy steel, Stellite on wearing surfaces and for trim. Material variations to suit service conditions.

DOUBLE SUCTION HOT OIL PUMP

DOUBLE SUCTION HOT OIL PUMP

(TWO STAGE)
Capacity Range: 1400-2000 gpm.
Head Range: 700-1100 feet.

Contained within a heavy duty, vertically split case with confined gaskets, this pump is a two stage unit, the first stage of which is double suction while the second stage is single suction. Initial entrance velocities have been materially lowered permitting operation at substantially lower npsh. Staggered volutes provide radial balance, while hub wearing rings on the second stage give axial balance. Stuffing boxes are extra deep and both stuffing boxes and bearings are water-jacketed. The rotating element can be easily and quickly removed as a unit without disturbing the piping or driver.

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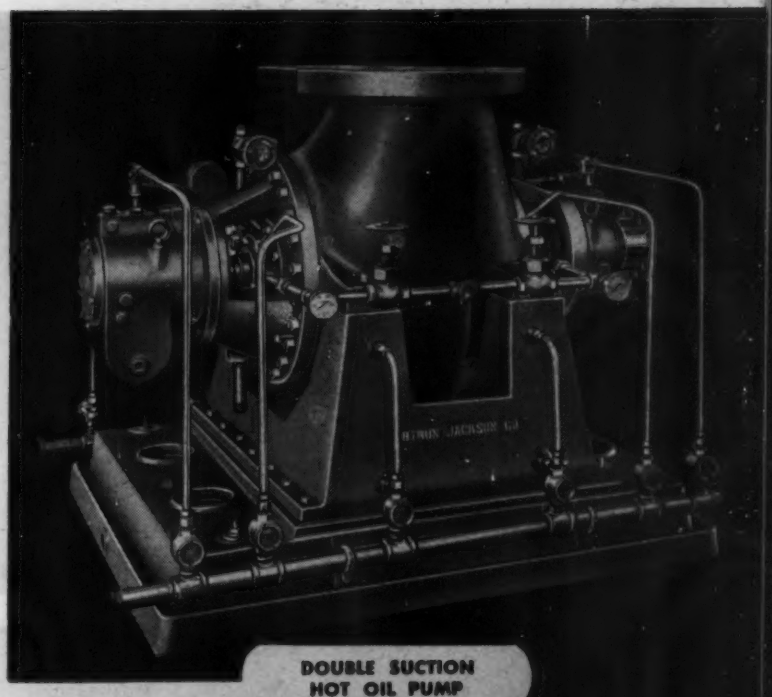
DOUBLE SUCTION HOT OIL PUMP
(SINGLE STAGE)

Capacity Range: 1000-7000 gpm.
Head Range: 100-700 feet.

Developed for capacities beyond the range of Single Suction Process Pumps. Design is based on proven principles of construction essential for superior service in handling high temperature liquids on continuous operation. Three pump sizes are available—6 x 8 x 10—6 x 10 x 14 and 12 x 16 x 20. Low entrance velocities and axial balance are obtained by use of a double suction impeller. Rotating element can be removed as a unit without disturbing the piping or driver. Pump can be constructed of a wide selection of materials to suit specific conditions.

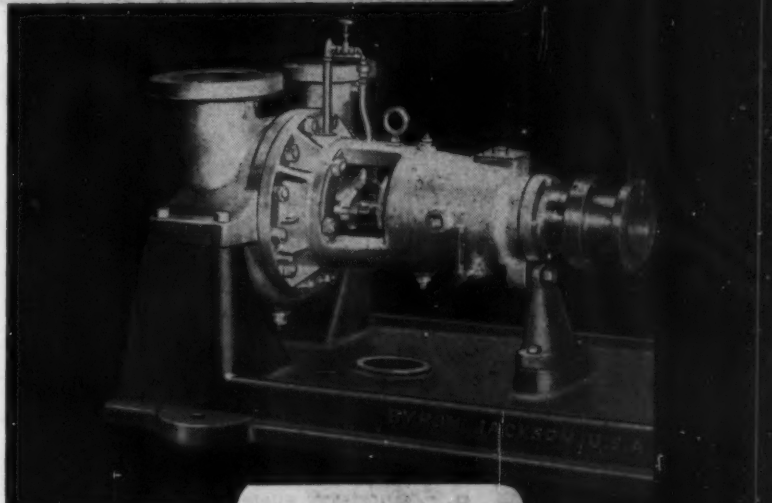


DOUBLE SUCTION HOT OIL PUMP

SM PROCESS PUMP

Capacity Range: 10-1400 gpm.
Head Range: 20-650 feet.

A horizontal, single stage, single suction, split case centrifugal pump for use where temperatures and pressures are high, suction liquid levels low, where hot and cold liquids near their boiling points must be handled, and where liquids impose severe conditions on stuffing boxes. An ideal pump for handling slurry. Alignment is maintained at any temperature. Bearing bracket, shaft and impeller assembly removable without disturbing piping, case, or driver. For temperatures to 850° F. Standard materials: Cases and impellers of cast iron, Meehanite, Ni-resist carbon or chrome alloy steel, or other alloy best suited to particular service.

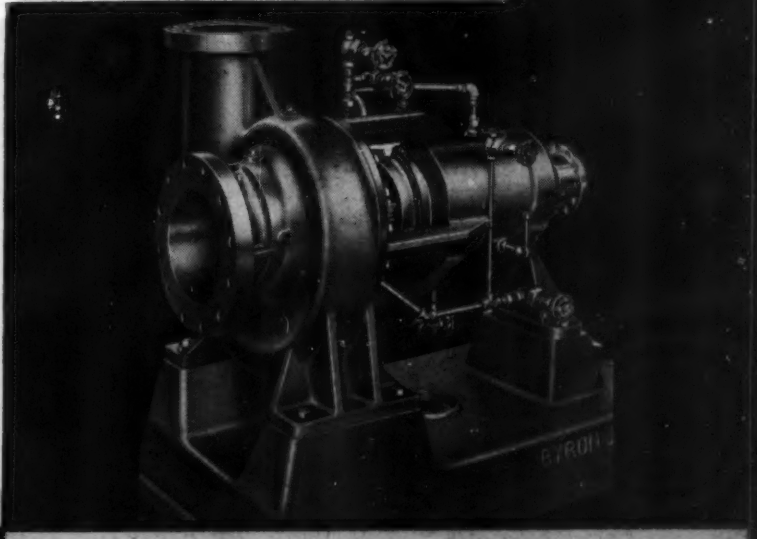


S. M. PROCESS PUMP

ACID BUTANE PUMP

Capacity Range: 2000-4500 gpm.
Head Range: 50-300 feet.

This special purpose pump is designed for handling acid butane and other corrosive liquids. It is a horizontal, single stage, single suction, split case centrifugal provided with a removable, split, water-jacketed, stuffing box for ease in packing. Impeller is arranged with wearing rings to obtain suction pressure on the stuffing box. Nozzles are adapted to conform to overhead piping. Bearing bracket, shaft and impeller assembly removable without disturbing piping, case or driver. Built in two sizes, 8 x 10 x 16" and 10 x 12 x 18". Materials to suit particular operating conditions.



ACID BUTANE PUMP

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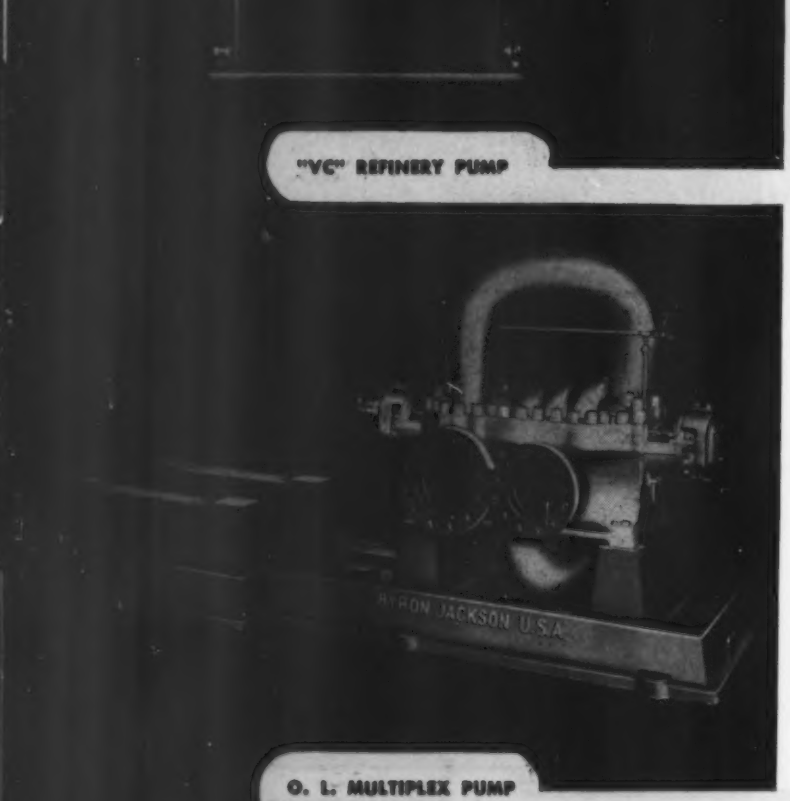
"VC" REFINERY PUMP

"VC" REFINERY PUMP

Capacity Range: 300-900 gpm.
Head Range: 300-1200 feet.
Temperatures to 900° F.

The "VC" Pump is a heavy duty horizontal, 2-stage, solid case centrifugal, fitted with single suction enclosed type impellers, mounted back to back to eliminate thrust. Designed for high temperatures and medium pressures. Suction and discharge nozzles, cast integral with the case, are vertically up to prevent gas binding. Entire rotating element may be removed without disturbing suction or discharge piping. Pump case is supported at centerline by cradle mounting to permit radial expansion in all directions under temperature without misalignment. Available in four sizes: 3 x 4 x 10, 4 x 6 x 10L, 4 x 6 x 10H and 4 x 6 x 12½. For installations requiring dual drive, double extended shaft can be furnished



O. L. MULTIPLEX PUMP

O. L. MULTIPLEX PUMP

Capacity Range: 250-2000 gpm.
Head Range: 600-2800 feet.

A horizontal, split case, multi-stage unit. Supplied with extended base plate and all metal flexible coupling, for direct connection to the driver Intended for medium capacity, high pressure service, this unit is particularly applicable for pipe lines, and other services requiring high pressures and high efficiencies. The case is made in two halves, which are bolted together on horizontal centerline by means of studs and acorn nuts. Suction and discharge nozzles are cast integral with the lower half of the case. The crossover passages, which are external, are cast integral with the case. Construction insures easy assembly. Standard materials. Cast iron, bronze fitted, nitrided Nitraloy or Stellite trim.

S. D. MULTIPLEX PUMP

S. D. MULTIPLEX PUMP

Capacity Range: 80-2000 gpm.
Head Range: 300-3000 feet.

A heavy duty, high speed, multi-stage pump, ruggedly constructed and adaptable for all classes of medium or high pressure service. The "SD" horizontal -split case unit is designed with crossover passages between stages cast integral with the case. This unit is always built with an "even" number of stages and with impellers paired and mounted back to back to equalize end thrusts. Staggered volute construction is employed. Includes sizes from 2" to 6" available as 2-4-6 and 8-stage units. Standard materials· Cast iron, bronze fitted.

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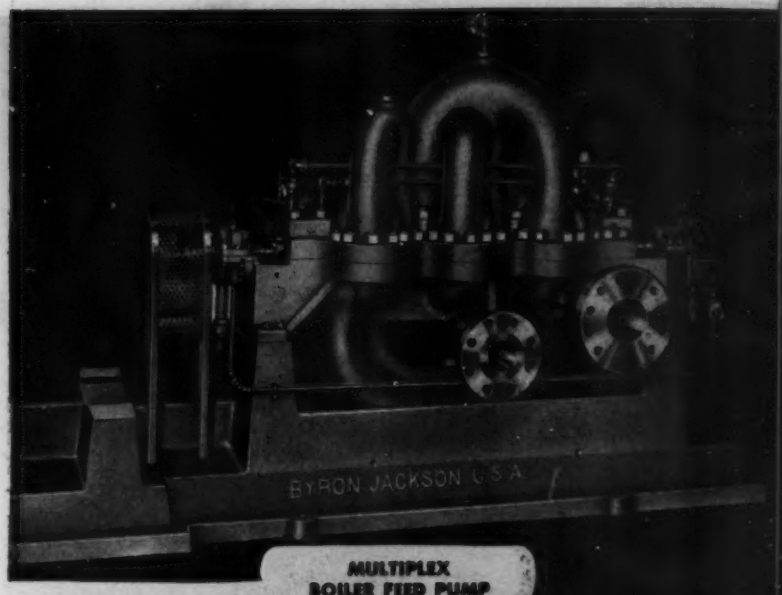
MULTIPLEX BOILER FEED PUMP

Capacity Range: 500-1100 gpm.
Pressure Range: 550-900 psi.

In general, this 4" x 6" 6-stage Split Case Boiler Feed Pump designed for medium capacity, high pressure service has the general characteristics of an OL Multiplex Pump, i. e., it is a horizontal, split case, multi-stage unit, designed with external piping to insure high efficiencies. However, construction, for this service, differs in the following respects. Volutes are staggered for radial balance. Stage arrangement provides for low pressure drop across bushings. First stuffing box is under suction pressure and second stuffing box is under first stage pressure only. A throttle bushing is furnished in the second stuffing box which permits bleeding back to suction pressure so that actually both stuffing boxes are under suction pressure. Base plate support is brought up under bearing brackets, providing a high mounting to permit free expansion.



MULTIPLEX BOILER FEED PUMP

FIGURE PUMP

Capacity Range: 10-1500 gpm.
Head Range: 10-300 feet.

The Figure Pump is a horizontal shaft, single stage, side suction centrifugal, mounted on heavy ball bearing bracket, with overhung shaft extended to receive flat or Vee-belt pulley. Designed for general utility pumping, it is a relatively inexpensive, single suction, general service unit. The design of these pumps incorporates rugged mechanical construction and hydraulic performance comparable to that obtained with double suction pumps of similar size. Production of these standard pumps in quantities permits the very nominal price at which they are offered. Sizes from 1" to 8" pressure range is to 200 lbs.

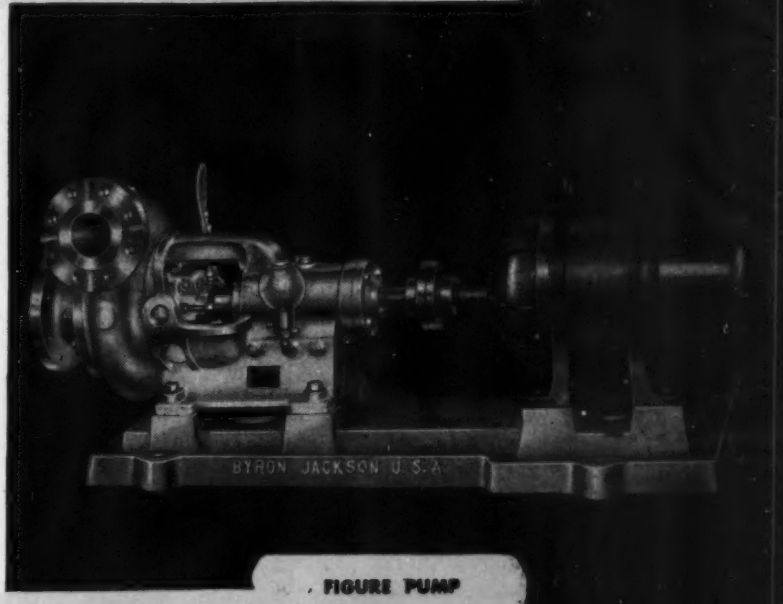
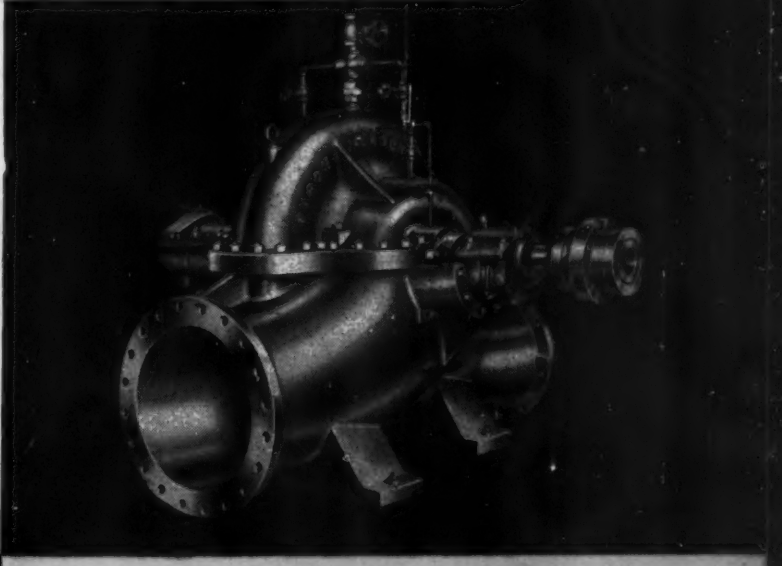


FIGURE PUMP

TYPE "S" PUMP

Capacity Range: 50-40,000 gpm.
Head Range: 40-400 feet.

A standard, all-round utility pump widely used by refineries for general cold or warm liquid pumping service, the Type "S" Pump is made in a complete range of sizes. Pumps presented under this classification are single stage, horizontally split, two-bearing type centrifugals. The pump is complete from suction flange to the discharge flange, including base-plate and flexible coupling for direct connection to driver. All impellers are machined on outside surfaces, hand filed in the water ways, statically balanced before mounting. Standard materials Cast iron, bronze fitted.



TYPE "S" PUMP

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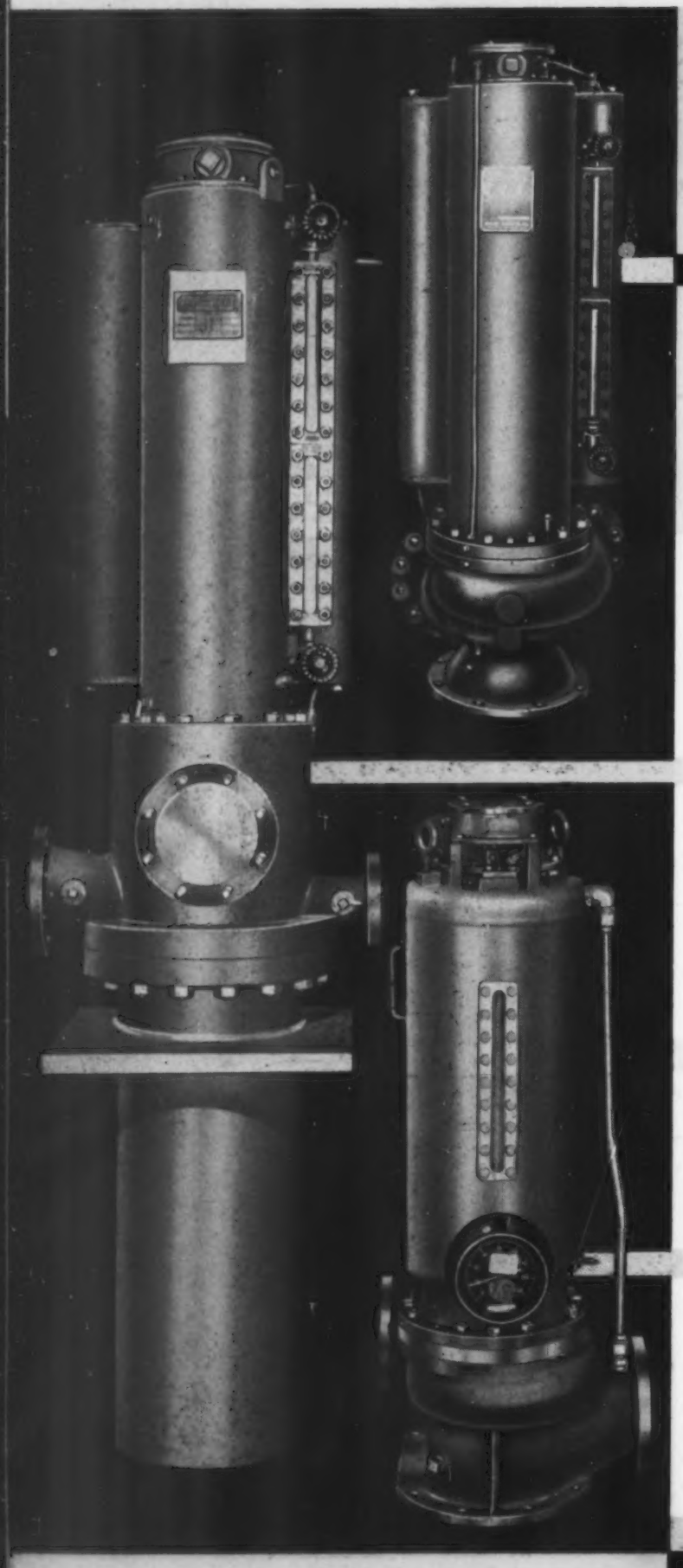

STUFFINGBOXLESS PUMP

Capacity, head and temperature ranges according to type of pump furnished for individual job application.

Basically different, **this pump has no stuffing boxes** and there is no escape to atmosphere of the pumped liquid. It is first choice for pumping highly volatile liquids. This pump is a combination of a 3-phase induction motor and any one of several standard type Byron Jackson pumps, built as compact vertical, self-contained, mechanically sealed, pumping units. The motor is built integral with the pump and is housed in a water-tight, jacketed casing which is filled with oil of high dieelectric strength to provide electrical protection and lubrication for the motor in addition to forming a medium for transfer of partial discharge pressure to the motor end of the mechanical seal. A two compartment oil tank assembly connected together at the bottom is also included as an integral part of the unit. An auxiliary oil supply is stored in the first compartment which is connected to the motor and the seal chamber. The second compartment is connected to a predetermined point in the pump case under partial discharge pressure. This compartment contains separating liquid (usually water) which isolates the motor oil in the first compartment from the pumped liquid in the second compartment. With the motor and oil tank assembly under partial discharge pressure, and the pump end of the mechanical seal under suction pressure, leakage from the seal is always to suction pressure—this leakage being replaced from the auxiliary oil supply. With this arrangement, low differential pressure can be maintained across the seals, regardless of suction and discharge as the pressure differential is determined by the point at which the pumped liquid is led from the pump case to the oil tank assembly. This differential will remain constant with varying pump pressures as any variations are immediately transmitted to the motor end of the mechanical seal through the oil tank assembly. The motor is 3 phase, either 220 or 440 volt, furnished in standard ratings from 5 HP to 125 HP inclusive at 2 pole speeds, and from 5 HP to 175 HP inclusive at 4 pole speeds. This motor is manufactured in its entirety by Byron Jackson Co. With the use of waterproof conduit, the complete unit can be operated completely submerged. At extreme left is illustrated a Hydropress Type Stuffingboxless Pump. At top left is illustrated a Type SM Process Stuffingboxless Pump and at lower left a Turbulence Type Stuffingboxless Pump.

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HYDROPLEX PUMP

Capacity Range: 15-55 gpm.
Head Range: 360-1600 feet.

The Hydroplex is a standard, low cost unit, designed primarily for handling non-abrasive and non-corrosive liquids. It is a vertical, single case, multi-stage pump, direct-connected to a 3550 RPM, 220/440 volt, 3 phase, vertical, drip-proof, solid shaft motor and sold only as a complete unit. Vertically split volute case and motor barrel are cast integral and bolted together with gasket between ground faces. Designed for low capacity, high pressure service. The vertical construction permits use of a simple foundation and requires a minimum of floor space. Standard materials: Cast iron, bronze fitted, chrome shaft. Maximum operating temperature is 250° F.; maximum operating pressure 670 lbs.; hydrostatic test pressure 750 lbs.

VMT PUMP

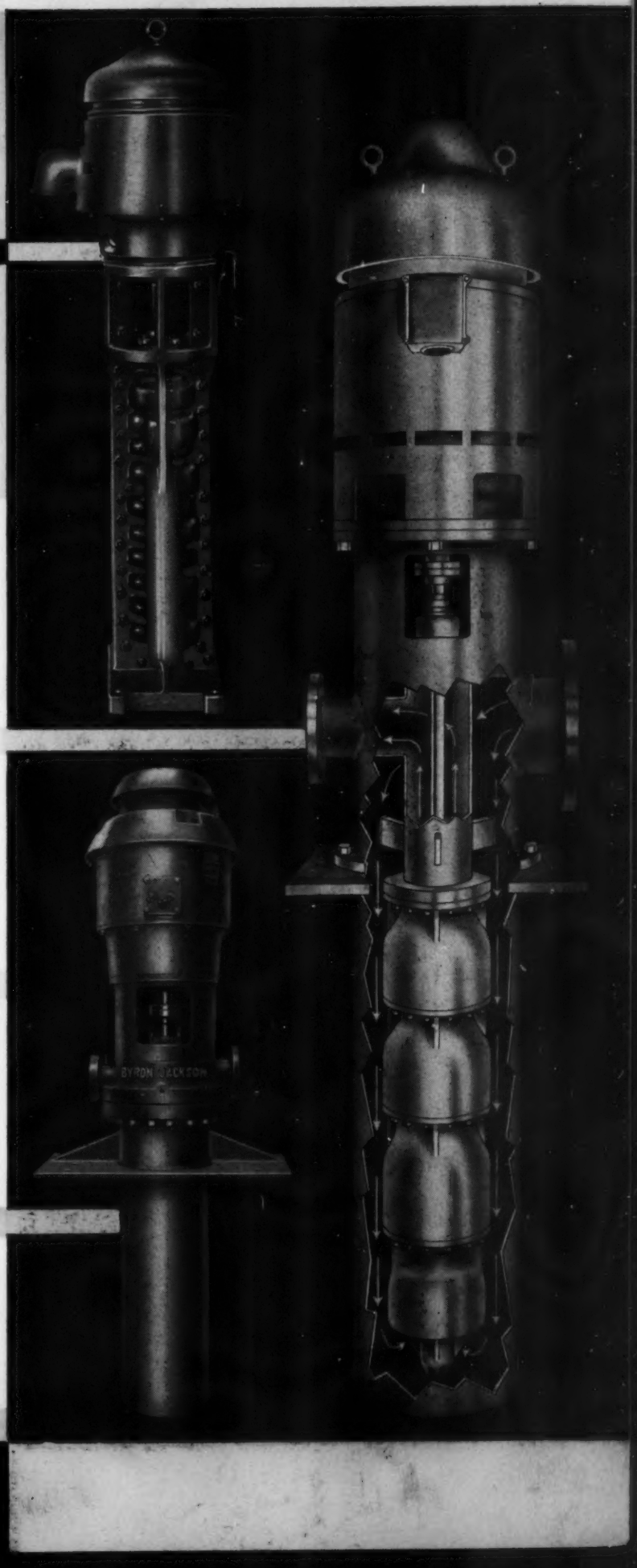
Capacities to 1000 gpm, heads to 250 psi, temperatures to 250° F, with standard materials. Larger sizes, higher temperatures on special order.

Designed for handling liquids—hot or cold—corrosive or non-corrosive—where the net positive suction head is limited. This pump is a multi-stage pumping unit mounted in a barrel from which it takes suction. Base of the pump is at ground level, while length of the barrel below is determined by the npsh requirement. This arrangement permits pumping from a suction vessel set at a lower elevation. Standard construction: Cast iron cases, bronze fitted, chrome steel shaft and shaft sleeve. Standard sizes. 8, 10, 12 and 14 inch barrels. Sizes to 5,000 gpm and for corrosive liquids and/or temperatures to 750° F., on special order.

HYDROPRESS PUMP

Capacity Range: 10-300 gpm.
Head Range: 200-7000 feet.

The Hydropress unit is vertical double case multi-stage pump, sold only as a complete unit with the direct-connected driver. Designed for high speed, low capacity, high pressure operation, the Hydropress unit offers many advantages. These include uniform nonpulsating flow, variable capacities without by-pass or speed change, elimination of relief valves. Vertical construction allows use of a simple foundation and requires a minimum of floor space. Standard materials: Electric welded steel or forged outer case; semi-steel inner volute case; bronze fitted with stainless steel shaft. Special materials for unusual service requirements.



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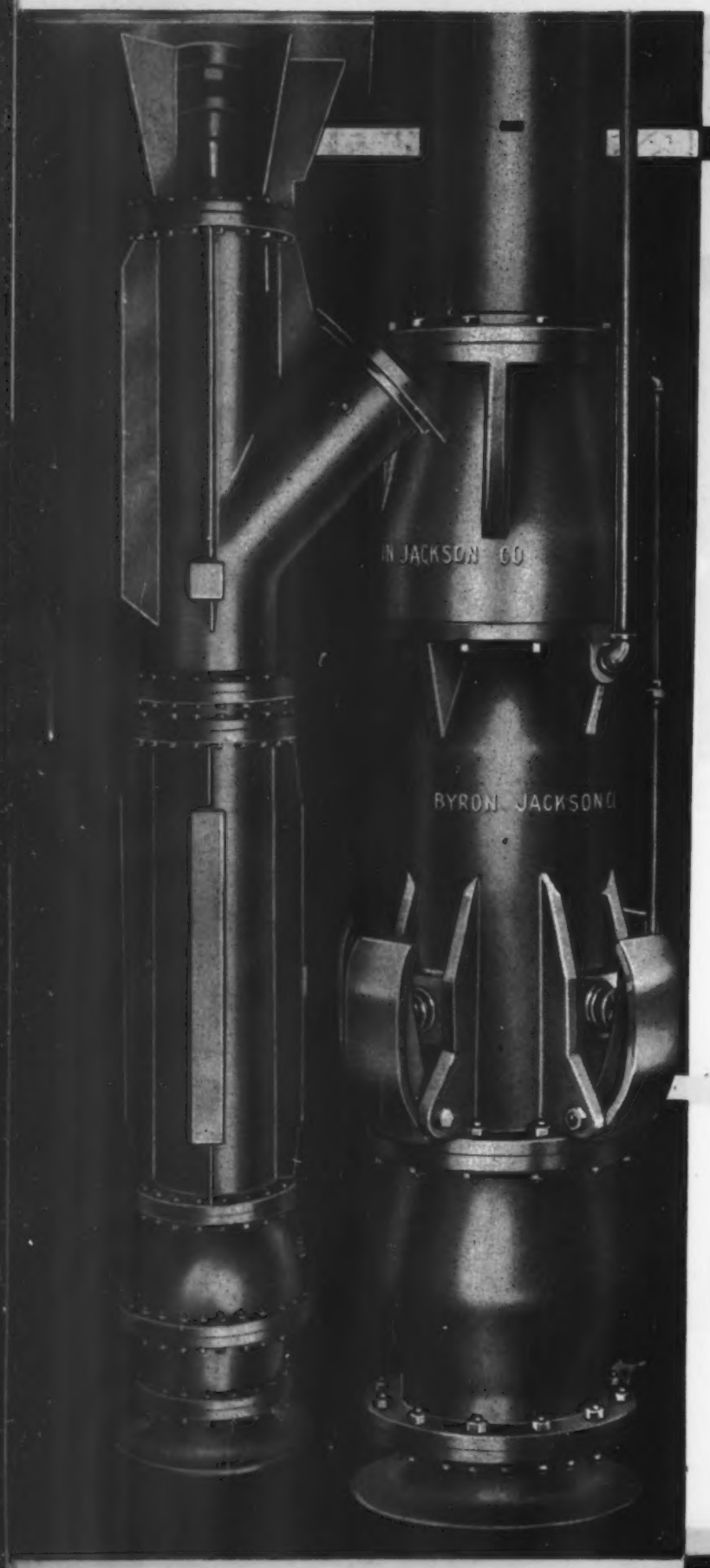

MOLTEN SALT PUMP

Capacity Range: 600-20,000 gpm.
Head Range: 20-70 feet.

A special purpose low and medium head propeller type pump for use in Houdry Catalytic Cracking units for handling molten salts having a temperature range of from 700 to 1000° F. This pump consists of a single or multi-stage pumping element suspended on a welded steel discharge column terminating at the upper end in a steel baseplate on which a motor or gear set is mounted to form a unit structure. Being of the submerged type, it is self-priming. Check valves, or other extraneous apparatus not required. The pump case, suction bell, and diffusion vanes are separate castings, bolted together. Combination suspension and discharge column consists of welded steel outer pipe in which the discharge elbow is incorporated. This unit is constructed of steel throughout and is heavily reinforced to prevent distortion from heat. Easily adapted to cracking unit and unusually long lived.

STRIPPER PUMP

Capacity Range: 600-2500 gpm.
Head Range: 30-400 feet.

Designed for close stripping of barges, tanks and/or other vessels this vertical pump handles gasoline and other light fuels in an ideal manner. This unit is a multi-stage type pump having a special priming stage located at the bottom and connected to the main pumping element by a vapor chamber. This priming stage is capable of pumping air or vapors. The pump is vertical and is installed in a welded steel well or barrel. Either vertical motor, gear or turbine drive can be used. The particular advantage of this pump is its self-priming ability. When unit is started liquid from the well is forced through the priming stage into the vapor chamber to act as a seal for the priming stage. After being sealed the priming stage operates as a vacuum pump exhausting vapors or air from the suction line and discharging them through the vent line into the cargo compartment or tank.

SALES ENGINEERING offices in New York, Chicago, Tulsa, San Francisco, Salt Lake City, Houston, Fresno, El Paso, Kansas City.

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P. O. Box 2017 Terminal Annex
LOS ANGELES 54, CALIF.

THE SUBMERSIBLE

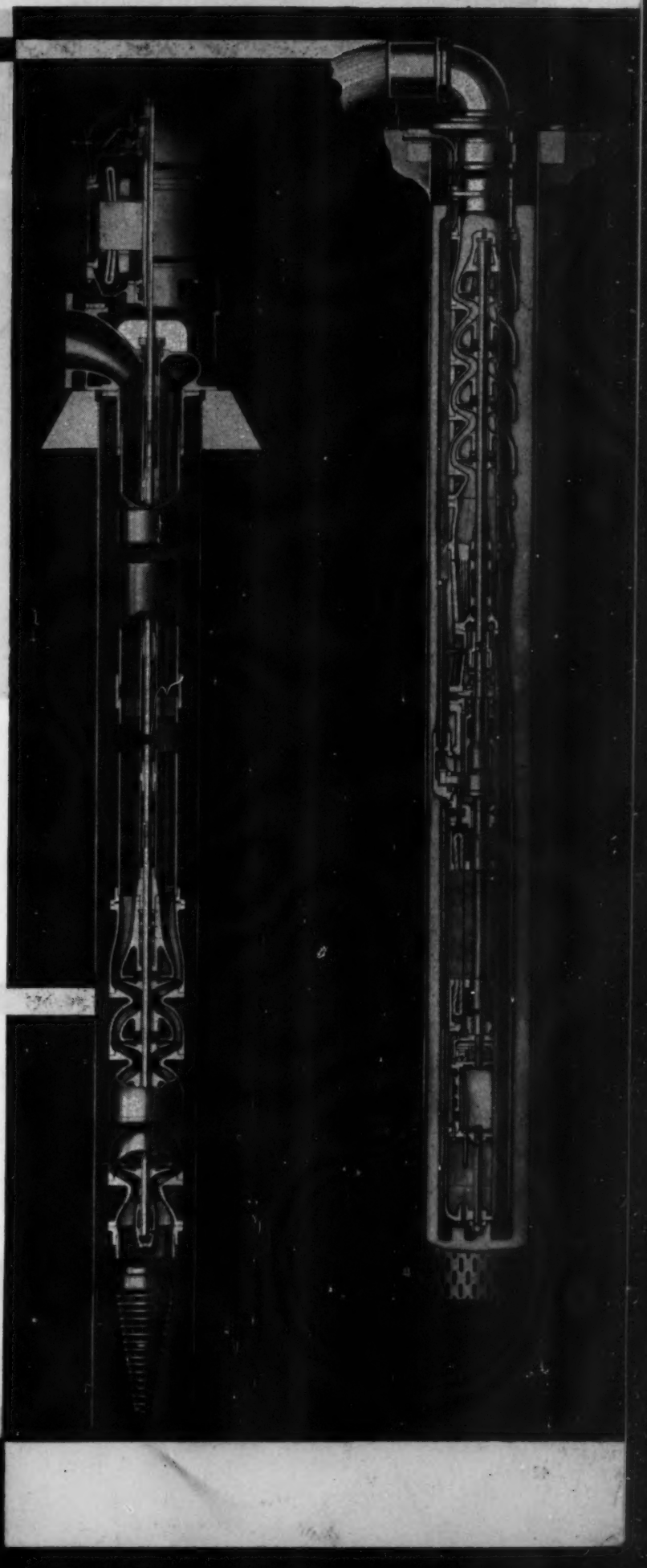
Capacity Range: 25-6000 gpm.
Motor Sizes: 5-350 hp.

A combination of a deepwell bowl assembly and a Byron Jackson vertical induction motor with patented features for sustained operation submerged in a few or under hundreds of feet of water. Motor is suspended below pump bowls and connected with them through a heavy cast iron grid-type strainer. Rotating element of bowl assembly is driven from the bottom where the extended shaft is coupled to the motor shaft by means of a steel coupling. Power is supplied to the motor through an armored submarine cable supported at 10 foot intervals on the column pipe and extending to starting equipment at the surface. Column pipe, from which motor and pump assembly are suspended, is supported at the surface by heavy combination surface plate permitting discharge elbow or vertical riser pipe to be attached. Ability of the Submersible to operate submerged is obtained through two major features—hydrostatic balance and a mercury seal. This pump is ideal for remote, concealed or difficult to reach locations.

THE DEEPWELL PUMP

Capacity Range: 25-7000 gpm.
Head Range: 10-600 feet.

Widely varying well diameters, capacities, heads, etc., are covered by three distinct types of bowl assemblies, each having different performance characteristics. Bowl assembly sizes are for installation in wells of 6 to 24 inches or larger. Several impeller designs are available for installation, for each size in each of the three types, providing more than 100 individual impeller designs of different hydraulic characteristics. Type "K" is designed for general use where average conditions prevail. Type "B" is designed for use where relatively high heads and pressures are involved. Type "R" bowl assemblies are designed for pumping large capacities from wells of restricted diameter. Three types of discharge heads are available — Standard, Heavy, and Extra-Heavy Duty. Head contains a discharge elbow; forms the base from which the pump is hung and provides a mounting for the driver. All discharge heads designed to mount either vertical hollow shaft motors; right angle gear drives; steam turbines; or pulley attachments, either flat or "V" belt types.



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The Cooper-Bessemer GMX and GMV are as modern as tomorrow's processing methods, completely in tune with the exacting demands of difficult or highly advanced processing work.

Weigh these GMX and GMV advantages:

VERSATILITY to power an exceptionally wide variety of compressor cylinders for any gas at any combination of pressure and volume within the horsepower range.

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Yes, your compressor requirements for today and tomorrow can be met ideally with Cooper-Bessemer V-angles, and with utmost economy and service satisfaction. Send for descriptive Bulletin No. 338 on the GMX, 200 to 400 hp; Bulletin No. 337 on the GMV, 400 to 1000 hp.

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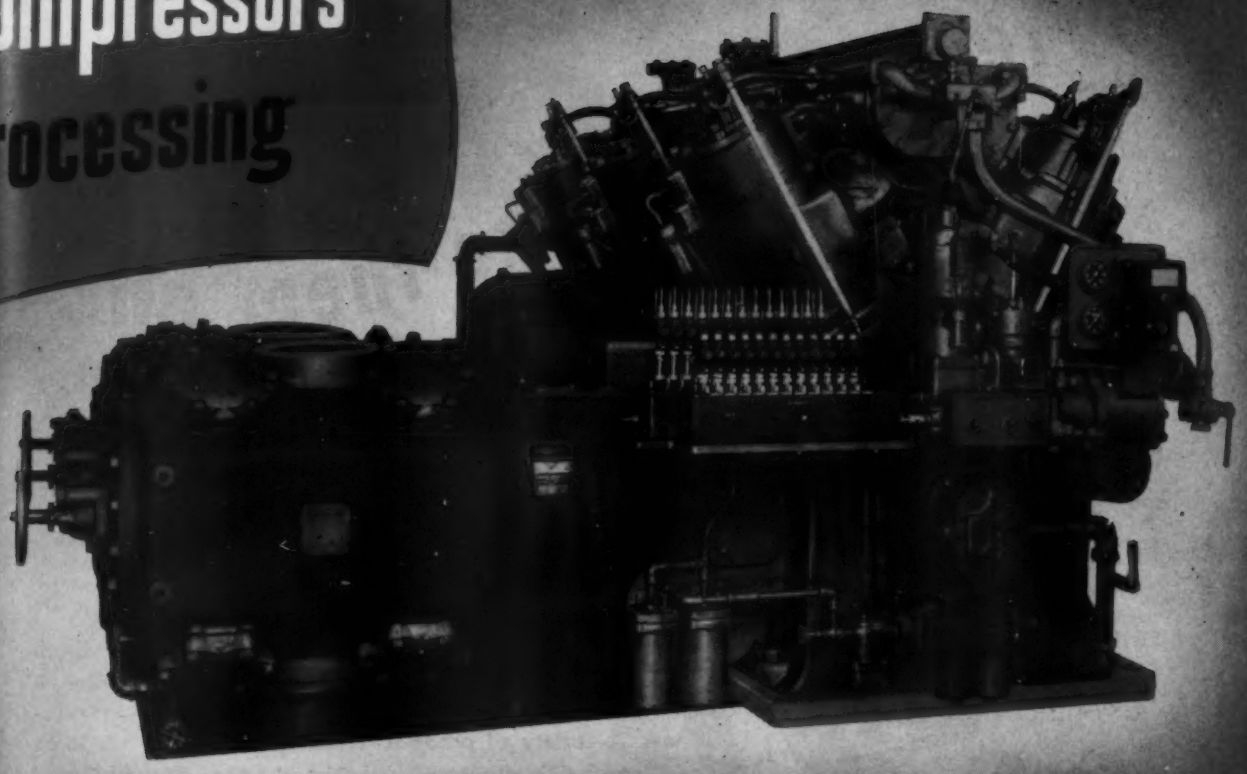
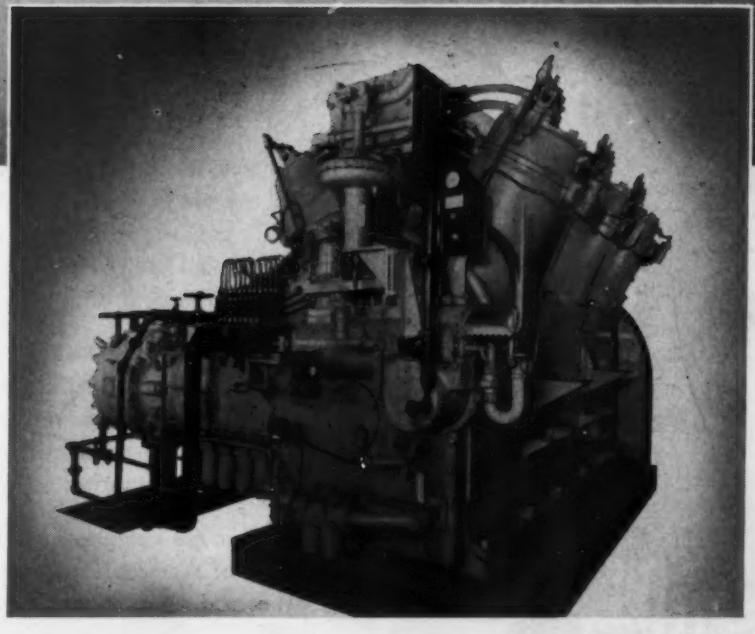
Compressors processing





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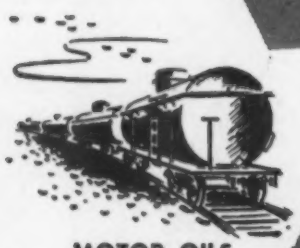
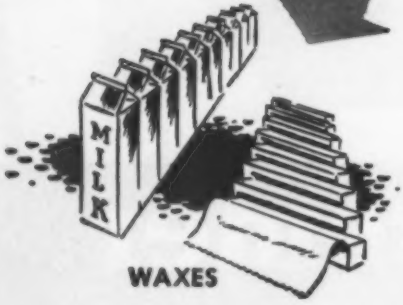
TURBINE OILS



MOTOR OILS



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The advantages of a consolidated filtering operation using a single, all-purpose adsorbent are becoming obvious to more and more refiners. A variety of products—ranging from white oils to heavy lubes—can be simultaneously percolated in one filter house. To do this may require a rescheduling of the operating cycles based on installed equipment—"order of the day" for Attapulgus' experienced engineers. What it *does* require is a flexible, efficient, across-the-board adsorbent—Attapulgus Fullers Earth.

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•

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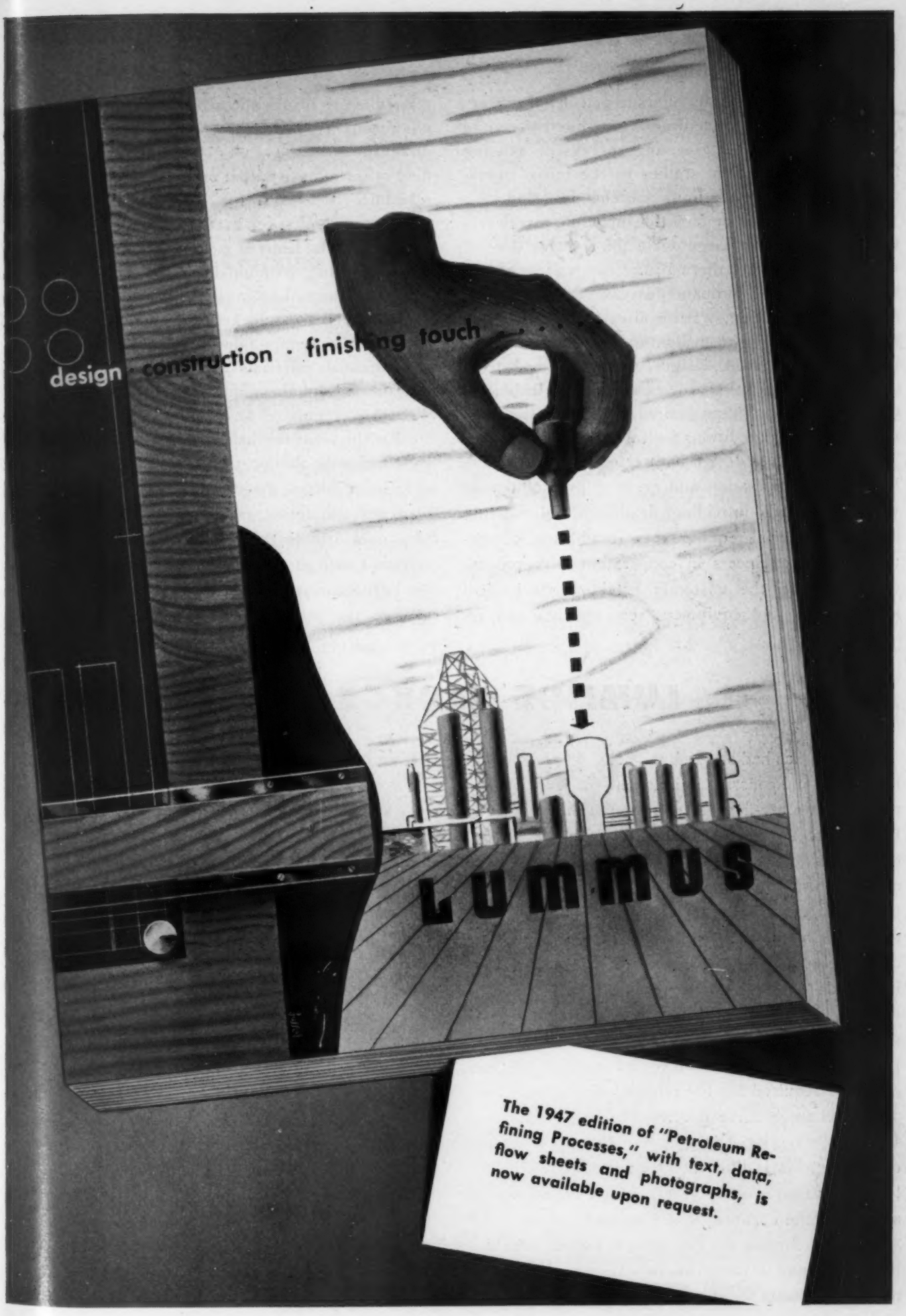

design · construction · finishing touch
LUMMUS
The 1947 edition of "Petroleum Refining Processes," with text, data, flow sheets and photographs, is now available upon request.

LUMMUS ORGANIZATION AND SERVICES

The Lummus Company offers to petroleum refiners and chemical manufacturers integrated services which extend from engineering surveys of existing facilities and economic studies to the initial operation of refinery process units and chemical plants.

Complete laboratory facilities including a wide variety of small and large scale pilot plants for all types of distillation, thermal and catalytic cracking, continuous coking, propane deasphalting, solvent refining and dewaxing, wax deoiling, and other processes are available for evaluation of stocks to establish all fundamental designs. Data obtained from pilot plant evaluations determine the yields and quality of the products desired as well as the optimum operating conditions for full scale plant design. Thorough process design is directed towards achieving optimum operation and greatest profitability as well as towards providing flexibility and meeting customer's special requirements. In addition, competent project engineers in cooperation with process engineers and the customer establish mechanical specifications and equipment requirements and arrangement to insure adequate performance of all this equipment as well as the entire plant, and finally direct the purchase of all material for shipment to the field in accordance with a predetermined construction schedule.

Lummus has specialized in the design and construction of the heart of most petroleum and chemical units—a heater distinguished by many advantageous design features. Lummus designs and fabricates all types of shell and tube heat exchange equipment, and operates a large pipe fabrication shop.

Fabricated materials are inspected in the shops and the flow of these materials to the field is expedited and regulated as much as conditions permit so that the plant can be constructed in a minimum of time and with the most efficient utilization of available labor forces. As construction of the plant approaches completion, experienced Lummus operating personnel field test the equipment and plant in accordance with customer's requirements, and supervise the initial operation leading to the customer's acceptance on the basis of prearranged performance tests.

LUMMUS BACKGROUND

Lummus has had some sixty years of experience in the design and construction of chemical plant process units and twenty-five years in the petroleum refining field. Since the birth of chemical engineering principles, Lummus has applied distillation, heat transfer, and flow of fluids in modern chemical and petroleum plant designs. Lummus has pioneered in the commercial development of a number of successful processes including lube oil solvent refining and dewaxing, selective cracking, polyforming, catalytic cracking, and wax deoiling, as well as ethylene, styrene, butadiene, phenol, and azeotropic toluene production units, and has designed and built all types of plants required for the refining of petroleum.

The comprehensive coverage in large volume of Lummus' experience in the petroleum refining and chemical fields is illustrated specifically by the following enumeration of plants which have been installed. In the chemical field Lummus has built over 300 units including alcohol, benzol, phenol, ethylene, butadiene, and styrene manufacturing plants. The number of plants for the manufacture of motor gasoline and 100 octane aviation gasoline from petroleum, including crude distillation, thermal cracking, catalytic cracking, polyforming and polymerization units exceeds 170. In the lubricating oil field, Lummus has built over 90 process units employing solvent refining and dewaxing, vacuum distillation, propane deasphalting and clay finishing processes.

Lummus performance includes quick delivery of completed plants with a record of long initial acceptance runs. Such records as twenty weeks from time of signing contract to complete on-stream operation for a solvent dewaxing plant, six months from time of signing contract to full scale operating performance for a two-stage atmospheric and vacuum topping unit in South America, and over thirty day initial runs for six consecutive thermal cracking units, as well as over two hundred days initial run for a catalytic cracking plant are some indications of Lummus performance. Lummus operations extend world-wide and include plants built in all parts of the world from the Netherlands Indies all through Europe and Russia and the Near East, South America and China.

GASOLINE MANUFACTURE

With gasoline production approximately 40% of crude run and quality ever on the increase, gasoline manufacturing processes represent the most important part of refinery operations.

Catalytic Cracking

Catalytic cracking was introduced in the petroleum refining field in 1938 and some one hundred plants were in operation with a total charge thruput of approximately one million barrels per day by the end of the World War II. Since then these figures have been increased in the order of 10%.

Catalytic cracking is being employed extensively for the conversion of heavy distillate fractions from crude oil into high octane number gasoline by once-through and recycle operations. This high octane catalytic gasoline in many refineries has become a major constituent of motor fuel blends and will become increasingly more important as engines designed for still higher compression ratios are brought into large scale production. Another important aspect of catalytic cracking is its ability to produce substantial volumes of home heating oil from feed stocks of higher boiling range while at the same time producing no residual fuel oil.

Virgin feed stocks for catalytic cracking units may be prepared by atmospheric and/or vacuum distillation, flashing in the presence of steam, or by propane deasphalting. Valuable additional quantities of gasoil feed stock may be derived from coking or viscosity breaking of heavy residual components of the crude which, if charged directly to a catalytic unit, would cause disproportionately large carbon deposition in comparison with the gasoline yield. Catalytic cracking units are capable of charging unvaporizable liquids as well as completely vaporized feed stocks. Single pass yields of 10 lb. RVP gasolines usually vary from 40-50% depending upon the charging stock, catalyst, and other process variables, while recycling yields of 10 lb. RVP gasoline may vary from 50-75% depending upon the aforementioned variables and the recycle ratio.

High Octane Gasoline Plants, Including Catalytic Cracking, Combination Thermal Cracking and Thermal Polymerization Units

GASOLINE MANUFACTURE (Cont'd)

Small capacity catalytic cracking units designed for thruputs ranging from 2000-6000 barrels per day are now available and afford the small refiner the opportunity to market gasolines of competitive quality. Such units have considerable flexibility as regards recycle ratio and cracking severity, as illustrated by a recently designed unit now under construction which will produce 72% of 10 lb. RVP gasoline having a clear ASTM octane of approximately 79. Catalytic cracking in general is characterized by high recoveries of liquid products with the absence of residual fuel oil production, high octane number gasolines, substantial yields of distillate suitable for home heating oil, simplicity of operation, and increasingly high on-stream efficiencies.

Lummus has built twenty-eight catalytic cracking units designed to process a wide range of charging stocks for the production of high octane aviation or motor gasolines. Many of these catalytic cracking units are supplied with synthetic bead catalyst manufactured in a plant designed and erected by The Lummus Company.

Thermal Cracking

Although catalytic cracking has become a major gasoline producer in many refineries, thermal cracking units of all types continue to play an important role in refining operations. Thermal cracking units designed to process stocks selected according to boiling range and refractivity produce the highest yield of high octane number gasoline. Lummus thermal cracking units incorporating heaters with a wide degree of firing control and with separate heating and soaking sections can easily accommodate the catalytic cycle stock and heavy reduced crude cracking requirements that have been brought about by the installation of catalytic cracking facilities. Lummus viscosity breaking plants are well adapted to process heavy residues to high yields of gasoline and low viscosity-low pour fuel oil through the use of vacuum reduction and cut-back of the asphaltic residue with light cycle stock. Lummus has designed and built sixteen thermal cracking units, most of which were three-coil and four-coil combination units with visbreaking, gasoil cracking and reforming coils. The performance record of delivery time and on-stream initial operation were outstanding for these units.

Polyforming

While the commercial history of the polyform process antedates catalytic cracking, this process continues to have an important role in processing of light hydrocarbons, naphthas, and gasoils for the production of high quality motor gasolines.

The polyform unit is a self-contained plant designed to process, in addition to the naphtha or gasoil charge, propane and propylene produced in the process and also butanes and butylenes, depending upon refinery vapor pressure requirements and other considerations. The charge to the plant may also include outside propanes and butanes. The fractionating system consists of high pressure fractionator, a stabilizer and an absorber which afford the means to supply the liquefied light hydrocarbon component of the furnace charge without requiring any compression or refrigeration. The presence of these light hydrocarbons in the furnace charge makes possible substantially more severe temperature and pressure conditions than can otherwise be achieved with the result that substantially higher gasoline octanes can be obtained. At the same time the temperature and pressure conditions are such that substantially all propane, propylene, butanes, and butylenes may be converted to gasoline. Propanes and butanes produced in the process may of course be supplemented with extraneous condensibles. The large circulation of light hydrocarbons through the system exerts a stabilizing influence on the overall plant operation which contributes to the characteristic ease of operation obtained in these plants.

Polyform gasolines are characterized by high research octane numbers and exhibit excellent road performance at all engine speeds. Naphtha polyform units charging a wide variety of heavy naphthas with and without extraneous propanes and butanes achieve substantially higher yields of higher octane number gasoline than is possible in conventional reforming units operating in conjunction with catalytic polymerization facilities. In general, naphtha polyforming affords a flexible means for adjusting plant octane number and vapor pressure requirements by means of its ability to simultaneously process extraneous surplus butane-butylene and low octane number naphthas. The polyform process is also well suited for the processing of refractory catalytic cycle stocks

requiring more severe cracking conditions than can be obtained in conventional thermal cracking plants. 10 lb. RVP motor gasoline yields ranging from 60-80% have been obtained from virgin gasoils, while yields ranging from 50-70% may be obtained from catalytic cycle stocks with octane numbers ranging from 72-76 for the virgin stocks and somewhat higher for the catalytic cycle stocks.

Lummus has built ten polyform units having a total circulation of 250,000 barrels per day, varying in size from 8,000 to 60,000 barrels per day of total circulation.

Polymerization Processes

In the field of gas polymerization, thermal and catalytic units which convert condensible light hydrocarbons to high octane polymer gasoline are available. The thermal polymerization process converts saturated as well as unsaturated hydrocarbons whereas the catalytic polymerization process polymerizes only the propylene and butylenes. Thermal polymerization gasoline yields range from 55 to 70% by weight according to the ratio of propanes and butanes, the degree of saturation and the operating conditions. Catalytic polymerization gasoline yields are substantially 100% by weight based upon the hydrocarbon disappearance, which amounts to more than 90% of the propylene and butylenes in the feed stock.

Reduced Crude Processing

The processing of reduced crude for the production of motor gasolines is largely dependent on the market demands for the various fuels. Thermal viscosity breaking which produces, in conjunction with further gasoil cracking, high yields of gasoline and marketable heavy fuel oil is frequently substituted by coking to further reduce heavy fuel oil production. Vacuum reduction and propane deasphalting with gasoil cracking are also used for the production of high yields of gasoline with low yields of heavy fuel oil. For the preparation of charge stocks for catalytic cracking units from heavy reduced crudes, the use of visbreaking, coking, vacuum flashing, and propane deasphalting must always be weighed from the point of view of carbon deposition on catalyst and life of catalyst. The processing of reduced crudes for lubricating oils is included in the section following.

Solvent Refining and Dewaxing Lube Oil Plants

LUBRICATING OILS AND WAX

With increased use of higher speed, higher compression automotive engines, as well as higher speed industrial machinery, the demand for better quality lubricating oils has gone hand in hand. The increasing trend towards the use of solvent deasphalting, refining and dewaxing, which had started prior to the war and which was postponed for four years, is now reestablishing itself.

A very popular lubricating oil processing system for maximum production of quality lubricating oils with minimum investment and operating costs, and maximum flexibility, includes vacuum distillation to low percent bottoms, propane deasphalting this minimum bottoms, single solvent refining the distillate cuts from the vacuum unit as well as the deasphalted oil, solvent dewaxing, and clay finishing of each of the blending stocks. Lummus has built a large number of complete lube oil plants incorporating these processes.

Vacuum Distillation

A recent Lummus complete lubricating oil plant included a two-stage vacuum unit for making raw distillate lube oil cuts which when solvent refined, dewaxed and clay finished, were suitable for blending directly into SAE grade lube oils. The features of this unit are the well fractionated cuts, low pressure drop in the towers, and the excellent color of the lube cuts. Lummus has built thirty-five vacuum distillation units for a wide variety of services throughout the world.

Propane Deasphalting

Propane deasphalting for separating asphalt and resins from residual lubricating oil by precipitation in propane solution may be carried out in counter-current settlers or by the use of a counter-current extraction tower. Propane deasphalting makes possible a higher yield of heavy lubricating oil from a reduced crude than is possible with vacuum distillation. Aviation lubricating oils may be produced by this process in conjunction with single solvent refining and solvent dewaxing processes.

Solvent Refining

Single solvent refining for removing selectively the objectionable constituents from charge oil and thereby improving the viscosity index may be applied to a wide range of stocks from very paraffinic through mixed base to extremely naphthenic materials. Single solvent refining units show the advantage of selective refining by ability to vary the solvent dose for the different raw vacuum distillate and deasphalted cuts. Lummus has built and has under construction twenty-two lube oil solvent refining plants with a total charge oil capacity of about 55,000 barrels per day and thruputs ranging from 350 to 6,000 barrels per day.

Solvent Dewaxing

Solvent dewaxing for removing wax constituents from lubricating oil stocks has been widely accepted for the production of specification low pour oils. While this process was well established before the war, many refinements such as stepwise injection of solvent with an attendant reduction in overall solvent requirement and continuous deoiling for wax production have been added. Some advantages of the process are the narrow spread in temperature between filtering temperature and pour of pressed oil as well as between the cloud and pour points. The process operates successfully on all types of waxy oils, either raw or solvent refined, over the entire range from spindle oils through cylinder stocks, and from low melting crystalline waxes to high melting point micro-crystalline waxes. Lummus jobs include thirty solvent dewaxing plants ranging in capacity from 400 to 6,000 barrels per day and with a total oil charge in excess of 55,000 barrels per day.

Emulsion Deoiling of Wax

The emulsion deoiling process for the reduction of high oil content slack wax (40-50%) in one-pass operation to 15% and in two-pass operation to 5% is finding application in the production of crystalline waxes. This partially deoiled wax is then charged to a conventional sweater thereby increasing the wax production capacity of this plant. Installations range in capacity from 200 to 2,500 barrels per day and have all been built by Lummus.

PETROLEUM CHEMICAL PLANTS

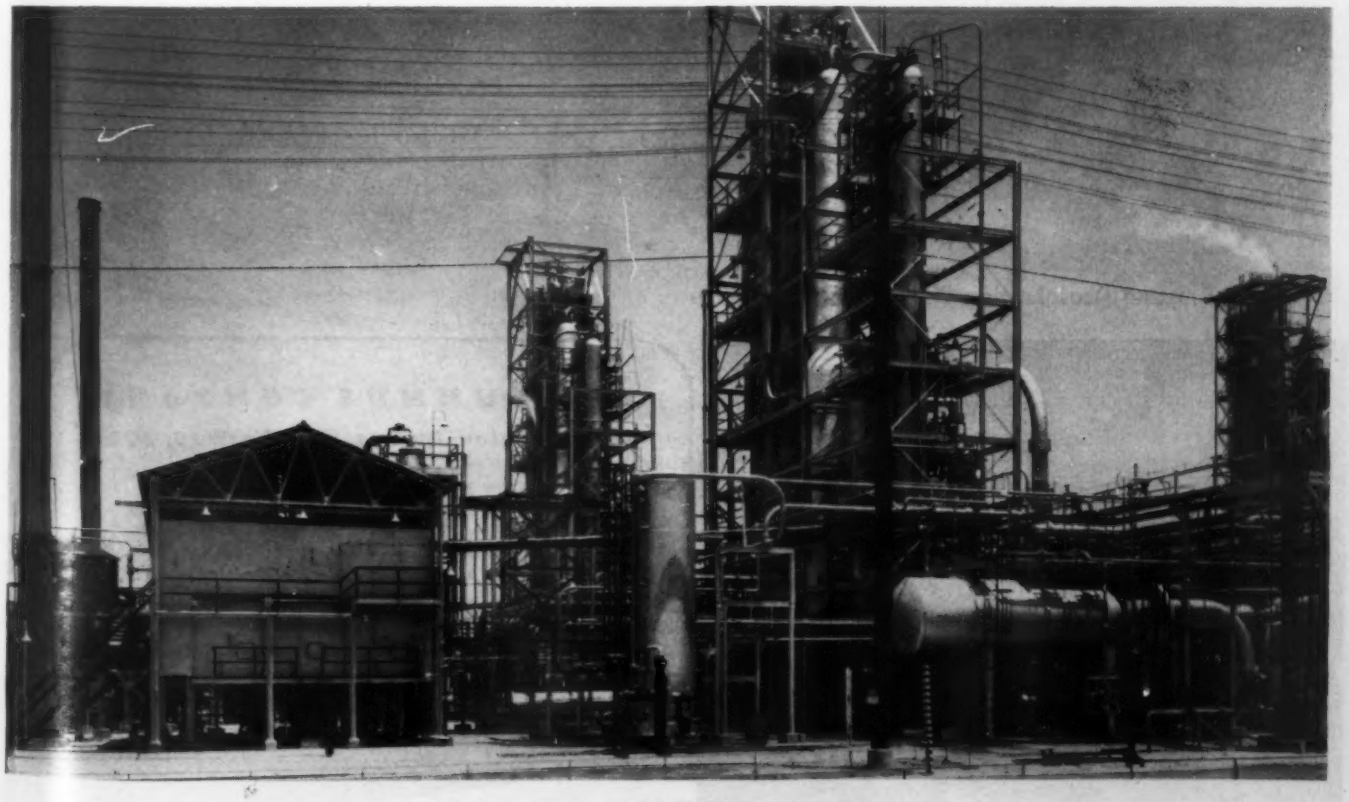
With the realization of the possibilities of petroleum as a cheap source material for the manufacture of many chemical products, the petroleum and chemical industries have joint interests. Petroleum is now employed in the production of many chemical products such as styrene, butadiene, phenol, toluol, alcohols, acetic acid, ketones, esters, resins, plastics, and solvents. In some instances the petroleum industry has gone into the manufacture of many of these chemicals, and substantial petroleum chemical manufacturing enterprises have been developed. In other situations the chemical industry has located large chemical manufacturing establishments convenient to oil refineries with which contracts have been negotiated for the supply of petroleum raw materials.

The development of petroleum as a source material for chemicals manufacture is accompanied by the development of new large scale manufacturing technique. This results in low overall manufacturing costs and therefore lower prices to the consumer. Although to date a substantial petroleum chemical industry has already been developed, this industry is on the threshold of further expansion, and excellent opportunities await progressive refiners and chemical manufacturers.

Typical of such operations is the manufacture of ethylene for use as an intermediate in the production of chemicals and plastics. Ethylene, and by-product propylene if desired, may be economically produced in large volumes by the pyrolysis of propane, ethane, and gasoils or reduced crudes. Lummus has designed four plants for the production of ethylene from petroleum having a total output of nearly half a million pounds per day.

The Lummus Company with a broad background of experience in the design, engineering and construction of chemical plants, petroleum refineries, and petroleum chemical plants is available for the development of projects for the production of raw materials from petroleum sources and the planning of process units for the manufacture of specific chemicals.

The three 50,000 ton butadiene plants built by Lummus have already demonstrated capacities in excess of 150%. Recoveries and purity of product have exceeded design expectations. These plants are capable of making low cost butadiene equivalent to 45 to 60 million passenger car tires per year.



Ethylene Plant, 100,000 Pounds Per Day

LABORATORY AND PILOT PLANT FACILITIES

The Lummus laboratories located in Bayonne, New Jersey, are well equipped with analytical, pilot plant and shop facilities and are staffed with experienced personnel for conducting all types of process investigaiton.

Complete analytical and small scale distillation facilities are available for crude oil, reduced crude oil and intermediate and finished product evaluations. The laboratory actively operates twenty continuous distillation systems, the largest capable of 5,000 gallons per day for atmospheric topping of crude oil, or 2,000 gallons per day for vacuum fractionation of reduced crude oils, and sixteen batch fractionation systems ranging from 25 litre capacity to a unique still capable of analyzing 1 cc.

The laboratory is equipped to establish fundamental design data for vacuum reduction, visbreaking, coking and propane deasphalting, all used for the production of cracking plant charge stocks.

Complete pilot plants are in constant use for full scale study of the manufacture of the entire range of lubricating oils. These include continuous units for atmospheric and vacuum distillation, propane deasphalting, furfural and phenol refining, solvent dewaxing and clay finishing. These pilot plants establish the yields, product quality and the design data required for the design, construction and operation of commercial plants.

The size of these pilot plants facilitates the preparation of large samples of raw, semi-processed and finished lubes for complete evaluation of commercial lube oil characteristics and full scale engine tests.

Lummus laboratories have conducted over the past half century extensive development work for the chemical process industries for such products as alcohols, acids and esters. More recently, Lummus has projected for the petroleum chemicals industry pilot plant work for such products as styrene, butadiene, phenol, toluene, resins, plastics and solvents into full scale commercial production.

A large proportion of the laboratory equipment is fabricated in the laboratory's own shop at Bayonne. This permits an opportunity for prompt translation of the process needs into semi-plant apparatus. The translation of semi-plant to commercial scale operations is facilitated by the employment of Lummus plant operators on the pilot plant equipment. A competent mechanical crew is constantly engaged in the building of new pilot scale equipment and in its modification and maintenance.

To amplify its petroleum services, Lummus has the active cooperation of licensor laboratories.

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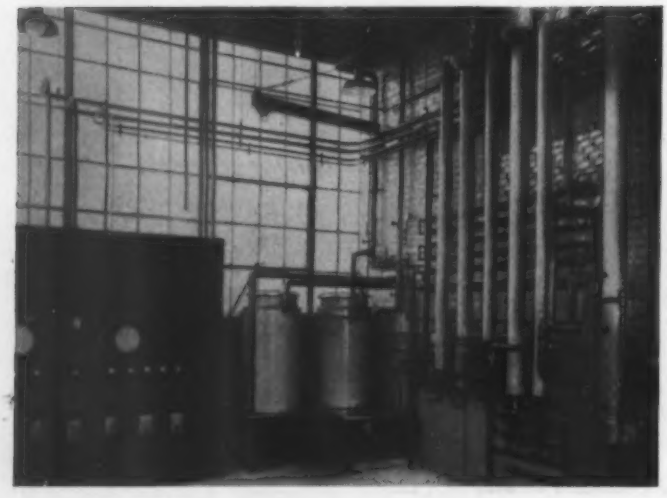
Propane Deasphalting Pilot Plant



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420 Lexington Avenue, New York 17, N. Y.

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long-life, low-cost, lube-oil plants

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PETROLEUM PROCESS REFINER HANDBOOK

Foreword

RESUMPTION of publication of the *Process Handbook of* PETROLEUM REFINER gives the industry an opportunity of evaluating its technical progress over the war period. Security dictated secrecy over a five-year period, with the result that this compilation of manufacturing processes makes an impressive total in comparison with the compilation of 1942.

Of more significance than the increase in totals, however, is the nature of this growth. For the first time petro-chemical processes are offered. The manufacturing division of the industry now has proof that both refinery and natural gases can be converted into chemical intermediates, accomplishments which have long been accepted as possible.

The other oustanding difference is the number of catalytic processes now available. It should be recalled that the first catalytic-cracking process was made public less than nine years ago. During the war period these processes fulfilled every prediction of the methods, while the application of this type of reaction came into vogue for accomplishments far removed from the cracking operation.

Another noticeable development is the readiness with which the industry already has shifted some of its new processes from wartime requirements into peacetime accomplishments. Ammonia from methane came into wide adoption as a source of explosive ingredients. The process now has application as a source of fertilizer ingredient. Equipment which provided toluene as another explosive ingredient now give industry its selected solvents. Styrene still serves the chemical-rubber program while it is providing the raw material for plastics.

Of the petro-chemical developments the conversion of natural gas into motor fuel is perhaps the most impressive. This issue of the *Process Handbook* for the first time gives some of the details by which the Fischer-Tropsch conversion has been adapted to natural gas as a starting material. Although this development doubtless leads industry's potential methods of providing assurance of motor-fuel supply, other new processes are of equal value from a scientific viewpoint.

Compilation of the processes would have been impossible without the cooperation of their licensors. Editors of PETROLEUM REFINER are grateful to the personnel of these organizations for help in the compilation. In most instances even the older processes are represented by revised flow diagrams and descriptions in keeping with recent development. The assistance of engineering staffs, which have provided the new drawings of the older processes as well as drawings for the processes now having their first publications, deserves especial thanks.

Acknowledgment also should be made of the suggestions that have come along looking to the improvement of the processes. These could not be incorporated for this issue, but they are available for adaptation as conditions justify in succeeding presentations of the processes that are serving the petroleum industry. Most of these diagrams as well as the descriptions were provided for only the *Process Handbook*. They are used with the understanding that reproduction is not permissible except by permission from the publisher as well as the process owner.

Absorption Oil Purifying System

PETROLEUM ENGINEERING, INC.

THE accompanying flow diagram shows an Oil Purifier Unit used in absorption plants, in the petroleum and coke-oven-gas industries. In the latter industry, absorption plants are used for extracting liquid aromatics from coke-oven gases.

This unit continuously removes suspended solids, waxes, gums and the higher mol-weight undesirable hydrocarbons from the circulating stream of absorption oil. Undesirable solids and heavy liquids accumulate continuously in the purifier unit and are periodically withdrawn from the system and discarded. The undesirable material is removed from the purifier unit, while hot, in form of heavy sludge, sometimes so viscuous it could not be removed unless hot. Ordinarily it has the consistency of road oil and frequently is used for that purpose around the plants. In coke-oven-gas plants withdrawal is frequent as compared to plants in the oil industry due to the nature of the gas processed. In natural-gasoline absorption plants, withdrawal may be every two weeks or possibly once a month.

In most plants it is possible to keep the absorption medium practically water white. This results in clean heat-transfer surface, oil coolers, heat exchangers and preheaters. Clean heat transfer apparatus means higher still and lower absorber temperatures, and improved efficiency.

Since heavier hydrocarbons are removed from the absorption oil, the result is a lower molecular weight, and therefore more efficient absorbent. Such an oil is easier to strip, so less process steam may be used or better stripping obtained with the same steam.

The purifier unit is operated in conjunction with the conventional still, may be considered a simple still, but it does not incorporate a condenser. The stripping section of the regular absorption-plant still serves as the condenser. Distillation is carried on at a temperature and pressure approximating the base temperature and pressure of the absorption still with which it works. The temperature is maintained by use of an internal heating bundle heated by steam or hot oil. All or the greater portion of the open process steam which is to be used in the plant still is passed through the purifier unit. Since the quantity of material evaporated in the purifier unit is relatively small as compared to the amount of material evaporated in the main still, the quantity of process steam passed through this unit is proportionately large. This means that absorption oil can be distilled under temperature and pressure conditions that would not be economical for separate distillation. Vapors from the purifier unit pass through a mist extractor and into the normal stripping steam connection of the absorption-plant still. These vapors are principally steam, but include a substantial quantity of oil vapors. The steam performs the usual function in the absorption-plant still and the oil vapors are recondensed on the stripping trays. In being recondensed these oil vapors give up heat at a point in the main still where the addition of heat is useful.

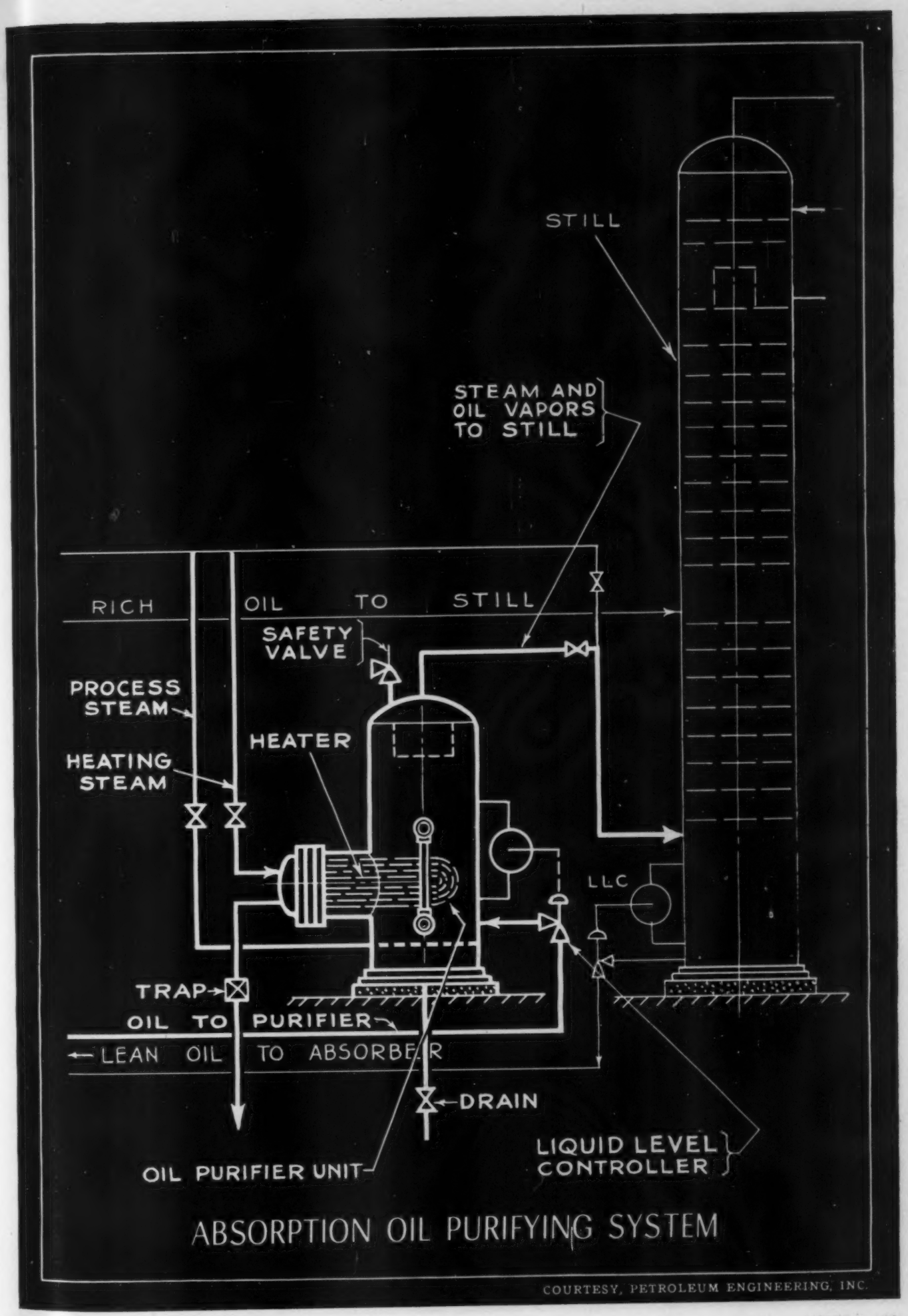
The quantity of oil distilled in this unit depends upon temperature and pressure, the quantity of the steam passed through the unit, and the physical characteristics of the oil remaining in the unit. Distillation rate gradually tapers off during the period of concentration of heavier materials in the purifier and it is the usual practice in most plants to dump the purifier unit when the distillation rate has been reduced to around 0.5 or 1.0 percent of the total plant circulation. Distillation rate can be checked by passing a sample of the purifier vapors through a condensing coil and comparing the quantity of water with oil condensed.

The only automatic control is a liquid-level controller, which maintains a level in the unit by admitting oil from the plant system as fast as oil is evaporated and released. No control is applied to the heating medium. If steam is used for heating, the full line pressure is allowed on the bundle, and the rate of distillation is not limited. In most plants, distillation rate is considerably higher than necessary for maintenance of absorption-oil condition. There is no reason however, for limiting the performance of the unit since its operation is advantageous to the operation of the main still in that heat is added to the stripping section.

No separate process steam is used. Whether steam or hot oil is used, the heat is recovered in the stripping section of the still. It may therefore be considered that the only extra plant utility required is heat lost in radiation from insulated surface. Heat radiation from the unit proper and hot piping that otherwise would not be installed in the plant, is negligible.

This oil purifier (patented) is designed and built by Petroleum Engineering, Inc., of Houston and Tulsa.

Since the initial tryout of this unit, this purifier has been incorporated as standard in absorption plants constructed by Petroleum Engineering, Inc., and several other plant designers. It also has been added to plants built before its inception in 1940.



ABSORPTION OIL PURIFYING SYSTEM

COURTESY, PETROLEUM ENGINEERING, INC.

Acetic Acid From Alcohol Process

E. B. BADGER & SONS COMPANY

A HIGH-STRENGTH crude acetic acid is obtainable from ordinary commercial grades of ethyl alcohol (180-190 proof) by this process. The crude acid produced is readily refined to glacial acetic acid. Some processes, such as esterification, can use the crude acid directly without further purification.

The production of glacial acetic acid consists of the following steps:

1. Oxidation of alcohol to acetaldehyde.
2. Acetaldehyde recovery.
3. Oxidation of acetaldehyde to acetic acid.
4. Refining of crude acid.

The alcohol-air mixture from the carburetor passes to the acetaldehyde generator where the alcohol is oxidized to acetaldehyde. The reaction is carried out in the vapor phase at elevated temperatures and in the presence of a silver-gauze catalyst. The acetaldehyde is absorbed in alcohol and then concentrated by distillation. Acetaldehyde then is fed continuously to the converter where it is blown with air. The reaction takes place in the liquid phase and at moderate temperatures in the presence of manganese acetate as a catalyst. The crude acid containing water, organic esters and a very small amount of catalyst, is refined to glacial acetic acid by distillation and evaporation.

It is well known that commercial ethyl alcohol can be produced utilizing ethylene recovered from refinery gases as the primary raw material. This process, therefore, makes the manufacture of either acetaldehyde or acetic acid from refinery gases a commercial possibility.

ACETIC ACID FROM ALCOHOL
ALCOHOL
AIR
ACETALDEHYDE GENERATOR
CARBURETOR
ACETALDEHYDE ABSORBER
ALCOHOL ABSORBER
STRIPPING COLUMN
ACETALDEHYDE COLUMN
ACETALDEHYDE
ACETIC ACID CONVERTER
ACETIC ACID COLUMN
EVAPORATOR
INERTS
WATER
STM
SOFT WATER
A.C.
FRESH CATALYST
SPENT CATALYST
LOW BOILERS
GLACIAL ACETIC ACID
COURTESY, E. B. BADGER & SONS COMPANY

Adsorptive Gas Dehydration Process

HOUSTON OIL FIELD MATERIAL COMPANY

ADSORPTIVE dehydration of gases by the use of granular desiccants is a recent development in petroleum processing. Adaption of activated bauxite to this service as here described follows the design of a unit by Houston Oil Field Material Company, Houston, Texas. It will serve in either field use or in plant use. In the latter the fuel for the furnace can be either gas or steam in keeping with plant operating economies.

The process, illustrated by the accompanying flow diagram, requires that wet gas first be run through a separator at controlled temperature and pressure, so droplets of oil and water can be removed and reduce the liquid load on the desiccant and lessen the chance of fouling it.

The gas enters the expansion chamber of the separator, where it is given a circular motion through a baffle plate covering the inlet. This motion gives the gas its initial centrifugal and impingement scrubbing. As it leaves this chamber it passes through a mist extractor where it gets a second and opposite centrifugal and impingement scrubbing. Liquid thus removed drains to the bottom of the separator.

From the separator the gas goes into the contactor, entering at the top and passing downward through a bed of activated bauxite, where remaining hydrates are removed. The contactors are designed with a diameter to give the gas a flow of 60 feet per minute while empty. By assuming a desiccant adsorption of 5 percent, 1 pound of water adsorbed per 20 pounds of desiccant, the quantity of bauxite necessary for each contactor can be determined. Contactor height is determined from contactor diameter and the volume of adsorptive material.

Contactors are so designed as to increase the height slightly in order to allow some free space between beds of the desiccant, which is supported on two trays with the free space between to counteract channeling.

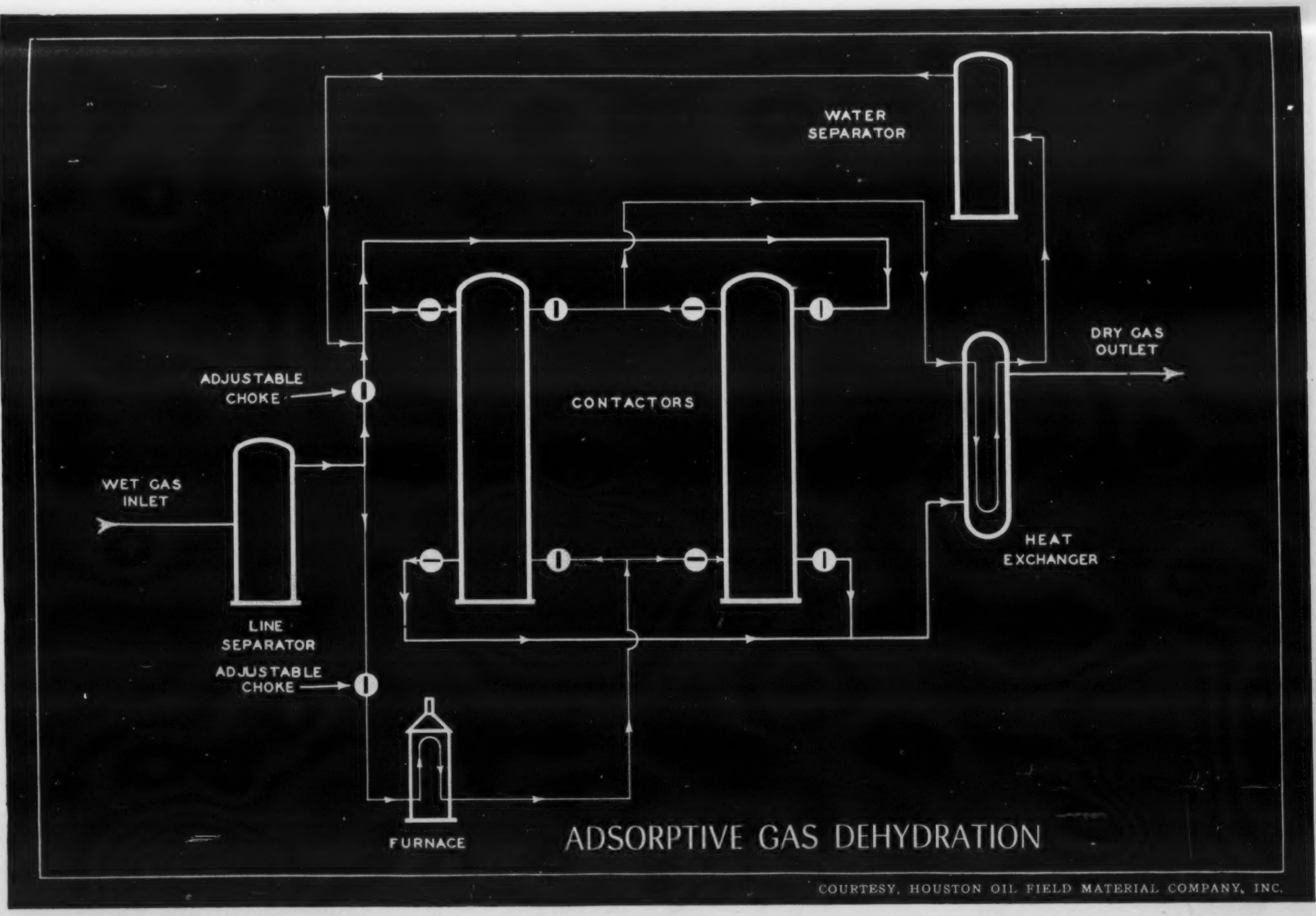
Both contactors and separators are constructed according to API-ASME codes for unfired pressure vessels, and are of multi-layer construction.

Dry gas passes from the contactor to plant use or to transmission line for distribution.

For flow control, an adjustable choke is used on the incoming gas stream between the line separator and the contactor. On the up-stream side of this choke, approximately 15 percent of the incoming gas is diverted by means of a second adjustable choke, so it can serve for regeneration purposes. The two adjustable chokes are used so as to assure proper pressure differential for regeneration. This diverted portion of the gas is heated to 450° F. and passed through the saturated contactor. This heated gas revaporizes the liquid adsorbed in the bauxite and carries it out of the vessel. Since the gas being dried passes through the bed from the top downward, its liquid content is higher upon entry. Consequently the regenerating gas enters the bottom of the bed and removed liquid at the top, thus reducing the time necessary for regeneration, since the liquid does not have to be driven through the whole desiccant bed.

From the contactor the regeneration gas passes through the tube section of the heat exchanger, where it is cooled by dry gas flowing through the shell. Cooled gas passes through a separator to remove liquid collected in the contactor, after which it is introduced into the incoming gas stream on the downstream side of the adjustable choke, with sufficient pressure to force it into the gas stream, so it can pass through the drying contactor with the wet gas and thus be fully dehydrated.

Constant temperature for regeneration is provided by having a recording temperature controller in the hot-gas line at the furnace and connected to a throttle valve on the fuel line to the burner. Another recording temperature controller is installed on the hot-gas outlet on the contactor and connected to a positive shut-off valve on the fuel line. When the outlet temperature on the contactor reaches 325° F. fuel to the furnace is automatically shut off, since this temperature is sufficient to remove moisture from the bauxite bed


WATER SEPARATOR
DRY GAS OUTLET
ADJUSTABLE CHOKE
CONTACTORS
WET GAS INLET
HEAT EXCHANGER
LINE SEPARATOR
ADJUSTABLE CHOKE
FURNACE
ADSORPTIVE GAS DEHYDRATION
COURTESY, HOUSTON OIL FIELD MATERIAL COMPANY, INC.

Alkylation Acid Regeneration Process

CHEMICAL CONSTRUCTION CORPORATION

THE CHEMICO Alkylation Acid Recovery Process is a development of Chemical Construction Corporation, New York. This method converts spent alkylation acid, regardless of its strength and carbon content, into new, water-white H_2SO_4 of any desired concentration. The system can also make additional acid from elemental sulphur or sulphur gases, either alone, or while spent acid is being reclaimed.

In brief, the process decomposes the spent alkylation-acid mixture by burning. The hydrocarbon content acts as fuel, and the acid itself breaks down into sulphur dioxide and water. The sulphur dioxide then is cooled, scrubbed, and passed into a contact acid plant, from which it emerges as new sulphuric acid, usually 98 percent H_2SO_4.

As shown in the diagram, the spent acid is pumped from a storage tank through an atomizing burner to the furnace, which is a brick-lined steel shell. The acid is decomposed at approximately 2000° F. with additional fuel or high oil sludges supplementing the hydrocarbons of the spent-acid mixture, if necessary. Preheated air is used for combustion. If desired, molten sulphur or hydrogen sulphide gas from other sources also can be burned in the furnace to supply heat and produce additional SO_2.

From the furnace, the gases pass to the recuperator, a tubular heat exchanger, and are cooled to 1200° F. The extracted heat is used to preheat the air fed into the furnace. From the recuperator, the gases go to the scrubbing tower. This tower is of steel, lined with sheet lead and acid-proof masonry, and is packed with spiral rings. Circulating dilute acid cools the gases to 250° F. and also washes out any dust which may have blown over from the furnace.

Next step is the cooling tower which is like the scrubbing tower, except it is not packed. The gases are cooled in this tower to 100° F. by water spray, and then go to the Cottrell mist precipitator. The SO_2 that dissolves in spray water of the cooling tower is removed in a stripping tower which is a lead-lined steel shell packed with spiral rings. A current of air passing through the water displaces the SO_2 gas which is returned to the main gas stream flowing to the precipitator.

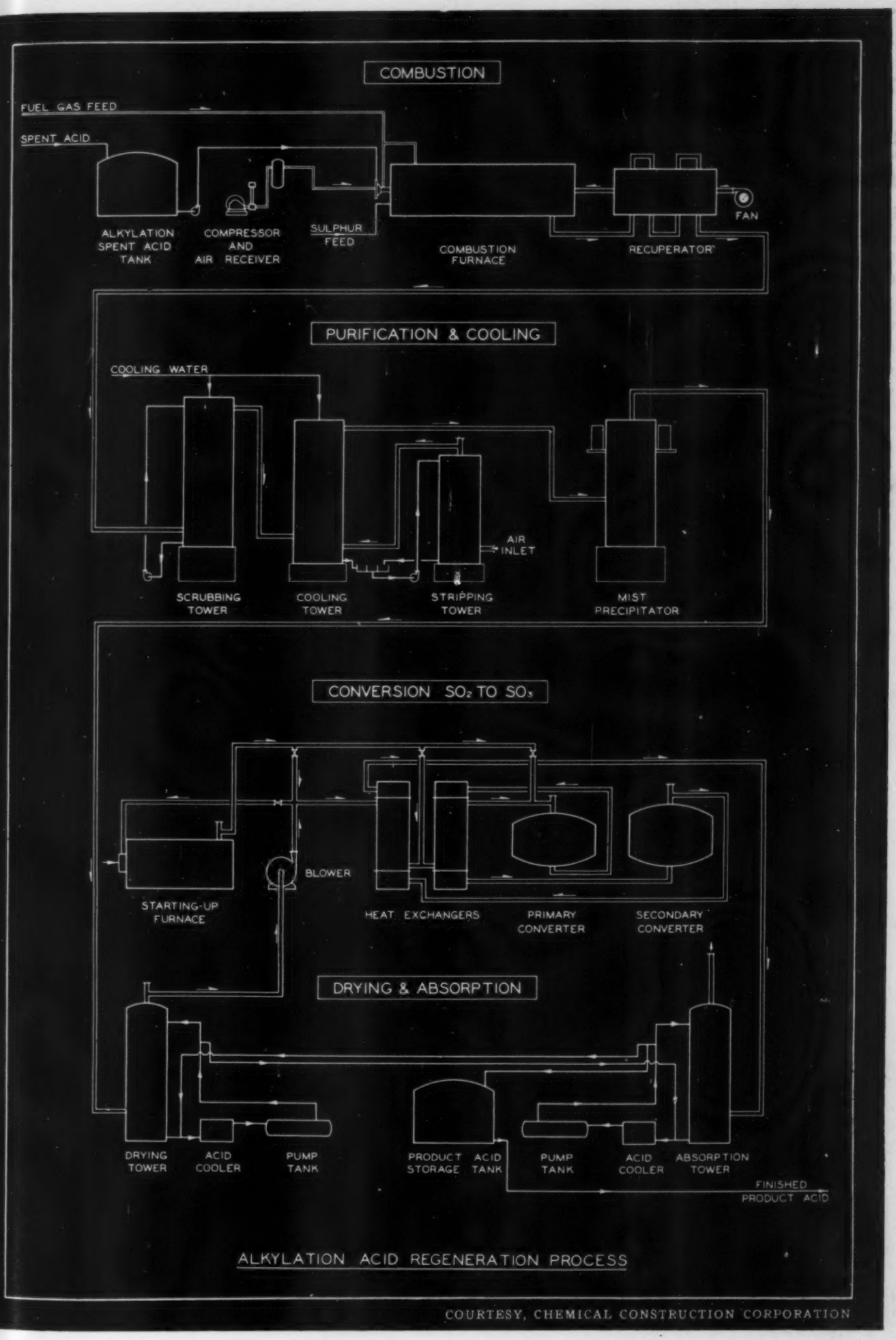
In the precipitator all the sulphuric acid mist in the gas stream is electrostatically removed and sent back to the scrubbing tower. The gas now is passed through a drying tower which has 93 percent sulphuric acid circulating over spiral ring packing. This acid is supplied from a pump tank and is cooled to desired temperature by cooling coils on its return to pump tank; sufficient 98 percent acid is constantly added to maintain the drying acid at 93 percent. As the dried gas leaves the drying tower air is added to reduce the SO_2 content to 8 percent, and the diluted gas is transferred through a centrifugal blower to primary and secondary heat exchangers for heating to 825° F.

Ready now for conversion, the heated gas enters the primary converter. This is an insulated steel shell containing layers of vanadium catalyst. The SO_2, in contact with the catalyst, is oxidized to SO_3, and rises in temperature to 1150° F. The hot gas is piped back to the hot side of secondary heat exchanger where it is cooled again to 825° F., the extracted heat being transferred to the incoming SO_2-and-air mixture entering the converter. The cooled gas goes now to the secondary converter and again is passed over vanadium catalysts to make sure all SO_2 is oxidized. This second oxidizing procedure generates more heat, which is removed by passing the gas through the hot side of the primary heat exchanger where the heat is absorbed by the SO_2 and air approaching the converters.

The SO_3 gas, at 250° F., now enters the absorption tower, a steel shell structure lined with acid-proof masonry and packed with spiral rings. The SO_3 is absorbed by the 98 percent acid circulating through the tower from the strong-acid pump tank and strong-acid cooling coils. Part of the 98-percent acid circulates back to the drying tower to maintain the drying acid at 93 percent and bring the absorbed water over to the absorption tower where it forms H_2SO_4 with the SO_3 gas being absorbed. Additional water is added as required.

The storage tank receives the newly formed 98-percent acid as it is drawn off from the strong-acid cooling system.

In getting the system started, heat is supplied to the converters by an external starting-up furnace.



ALKYLATION ACID REGENERATION PROCESS

COURTESY, CHEMICAL CONSTRUCTION CORPORATION

Ammonia From Natural Gas

LION OIL COMPANY

CONVERSION of natural gas into ammonia came into wide application during the war period as a source of explosive ingredients. One of these plants, Ozark Ordnance Works was operated by Lion Oil Company, El Dorado, Arkansas, and has since become a peacetime activity of the company, which now produces anhydrous ammonia, aqua ammonia, ammonia-ammonium nitrate solutions as well as ammonium nitrate fertilizer, from natural gas, air and water.

This process description and the accompanying flow diagram were provided by the Chemical Division of the company and represent expanded operations of the war-built plant. From a product viewpoint the process consists of three parts: manufacture of anhydrous ammonia, manufacture of nitric acid, and the finishing of fertilizer.

Steps in the manufacture of anhydrous ammonia include preparation of the raw (suction) gas at the reform plant, purification of the suction gas, and fixation of the purified hydrogen-nitrogen mixture in the synthesis operation to form ammonia. Reforming of natural gas is accomplished by passing a mixture of gas and steam downward through tubes and over a nickel catalyst at 725° C. Air is added to furnish nitrogen, the oxygen being consumed by hydrogen, and simultaneously a portion of the residual methane is converted into hydrogen by secondary reforming. The water-gas reaction carried out in the CO converter in the presence of iron oxide catalyst then accounts for the remainder of the hydrogen produced in the reform step.

Suction gas containing approximately 20 percent impurities (CO_2, O_2 and CO) is first scrubbed with water at 225 pounds pressure to remove most of the carbon dioxide. Then the gas is scrubbed with ammoniacal copper ammonium formate-carbonate solution and then 5 percent caustic at 1800 pounds pressure for removal of remaining carbon dioxide, all of the oxygen, and all but a trace (10 ppm) of carbon monoxide.

In the synthesis loop the purified process gas (make-up) and recycle gas both enter the oil filter at 5000 pounds pressure, pass to the cold exchanger, thence to the secondary or ammonia-cooled condenser, back to the cold exchanger, and then to the ammonia converter where, by exchange with the hot exit gases, the inlet gas is heated and enters the catalyst bed at 450° C. Passing through the catalyst, fixation of the hydrogen and nitrogen takes place at 5000 pounds pressure and a hot-spot temperature of 525° C. Only a portion of the hydrogen-nitrogen is converted, the converter inlet gas containing 2.5 percent ammonia and the outlet 12.5 percent. An iron catalyst promoted with aluminum and potassium oxides is used in the conversion of the hydrogen-nitrogen mixture to ammonia. The gas passes from the converter to the primary or water-cooled condenser, from which it enters the primary separator, and then goes to the circulator for recompression, and delivery of the recycle gas to the oil filter. Anhydrous liquid ammonia is continuously drawn off the cold exchanger and primary separator.

The process for manufacturing nitric acid comprises three steps: ammonia oxidation, secondary oxidation and absorption. In the first step an ammonia-air mixture at 60 pounds pressure is passed over a hot platinum-rhodium gauze, maintained at 900° C., and burned to nitric oxide (NO). The gases are cooled and then pass to the oxidation tower where the introduction of secondary air produces the nitrogen dioxide (NO_2). This material then is absorbed in water by means of a system of absorption towers, circulating pumps, and trickle coolers to form 56 to 57 percent nitric acid.

In the manufacture of the ammonium nitrate fertilizer nitric acid (56-57 percent) first is neutralized with anhydrous ammonia to form an 83-percent ammonium nitrate solution. This solution is concentrated in a vacuum evaporator and the melt charged to a pelletizing tower. The pelleted material is removed, dried, cooled, and coated to produce the desired storage and drillability qualities. The latest design which industry affords is incorporated into the process and a superior product is obtained.

A unique feature in the design of the Lion plant is that natural gas is used throughout as the prime source of power. Such includes its use as fuel in the primary reform furnaces, as fuel at the steam plant, and as fuel for driving gas engines. These gas engines, 54 in number, are housed in the compressor building and represent nearly 60,000 horsepower. All the engines are 1000-horsepower each with the exception of 11 1000 K.W. engines used on electric-power generation. Twenty-four of the engines are used on process gas compression, 5 on refrigeration, 6 on air compression for nitric acid, and 8 for driving water pumps in the water scrubbing operation.

The chemical plant is operated as a division of Lion Oil Company.

AMMONIA-NITRIC ACID PROCESS
COURTESY, LION OIL COMPANY, CHEMICAL DIVISION
GAS REFORMING PLANT
AMMONIA PLANT

Azeotropic Fractionation Process

UNION OIL COMPANY OF CALIFORNIA

AZEOTROPIC fractionation is a special type of distillation process adapted to the separation of a mixture of compounds that can not be readily segregated by the more conventional distillation procedures. Such mixtures may be composed of either (1) components having essentially the same boiling points such as heptane, boiling point 98.5° C., and methyl cyclohexane, boiling point 100.9° C., or (2) components that form azeotropes among themselves such as cyclohexane and benzene which give a constant boiling mixture (azeotrope) distilling at 77.7° C. The usual petroleum fractions encountered in normal refinery practice represent a combination of both of the above conditions. In many cases, essentially pure compounds can be recovered from such mixtures by adding a carefully chosen material, termed an azeotrope former, which will alter the relative vapor pressures of the components in such a manner that upon distillation one of the components is obtained separately from the others.

The production of nitration-grade toluene from petroleum fractions, an operation which was conducted on a large scale during the war, is a typical example of the advantages of azeotropic fractionation. Toluene occurs naturally in the distillates from certain crudes and the quantity can be considerably increased by the catalytic treatment of such distillates. However, even under the best of conditions when applying conventional distillation procedures, the maximum toluene concentration which can be obtained is the order of only 70-90 percent, depending upon the recovery desired. On the other hand, by adding a mixture of methyl ethyl ketone and water to these toluene-containing distillates, it is a relatively simple matter to recover toluene of 99+ percent purity with yields in excess of 95 percent. The accompanying flow diagram, detailing the recovery of toluene from petroleum fractions, is illustrative of azeotropic - fractionation technique generally.

Referring to the flow diagram, the toluene-containing fraction and the azeotrope former, a mixture of methyl ethyl ketone (MEK) and water, are introduced into the azeotroping column. The constant-boiling mixtures of non-aromatic hydrocarbons, water and MEK pass overhead, giving a bottoms of substantially pure toluene. However, in order to meet Army specifications for nitration-grade toluene, a light acid treatment is required and the acid-treated material then is rerun in a finishing column to give a product of the desired quality. The overall recovery of toluene, based on the toluene present in the feed stock, is above 95 percent.

The overhead product from the azeotroping column is washed with water to recover the MEK. The dilute MEK solution from the extraction tower passes to a recovery column where the MEK-water mixture used as the azeotrope former is distilled overhead and recycled to the azeotroping column. The water from the bottom of the recovery column recycles directly to the extraction column.

While the azeotropic process has been described for the segregation of toluene from a petroleum fraction the same general procedure is applicable to the treatment of many other mixtures such as the recovery of benzene and xylenes from petroleum fractions, the separation of aromatic hydrocarbons from thiophenes, paraffins from naphthenes, alcohols from ketones, etc. Detailed information on specific problems can be obtained from Union Oil Company of California, Los Angeles.

AZEOTROPIC DISTILLATION PROCESS
PETROLEUM FRACTION
200-240° F. ASTM
METHYLETHYLKETONE (MEK) AND WATER AZEOTROPE FORMER
MEK-WATER NONAROMATICS
NONAROMATICS
WATER
MEK-WATER EXTRACT
WATER
TOLUENE 99%+
ACID TREATMENT
CAUSTIC NEUTRALIZATION
WATER WASHING
AZEOTROPING COLUMN
EXTRACTION COLUMN
AZEOTROPE RECOVERY COL.
FINISHING COLUMN
NITRATION GRADE TOLUENE
HIGH BOILING POLYMERS, ETC.
COURTESY, UNION OIL COMPANY OF CALIFORNIA

Butadiene Process

STANDARD OIL DEVELOPMENT COMPANY

RAPID development of commercial processes for the manufacture of butadiene resulted from the requirements of the wartime synthetic rubber program. Butadiene by the catalytic dehydrogenation of normal butane or butylene previously had been the subject of considerable research and pilot-plant work. In going from butane to butadiene it appeared that there would be some advantage in operating the process in two stages, while from butylenes a single-process stage was possible. Wartime demands of butylenes for gasoline production by alkylation and polymerization processes were heavy, but large-scale commercial development of catalytic cracking promised sufficient yields of unsaturates to meet requirements, and finally determined the choice of the one-stage route, using butylenes as the feed stock.

In the catalytic dehydrogenation of butylene, the principal process problem centers about the reactor. The design is complicated from the engineering standpoint by the requirements imposed by the chemical nature of the reactant and product, and by the thermodynamics of the reaction. The most serious limitation in process design is the tendency for butadiene to polymerize and ultimately to degrade to coke and hydrogen. This can be minimized by operating at hydrocarbon partial pressures of the order of 90 to 100 millimeters. The low partial pressures can be attained by low-pressure operation or by the use of an inert gas as a diluent.

To obtain reasonable butylene conversion, reaction temperatures above 1100° F. have been found necessary, but to avoid thermal cracking of the butylenes in heaters and transfer lines temperatures should not be in excess of 1300° F., so that for practical purposes the operating temperature range is 1150° to 1250° F. at a superficial reactant residence time of about 0.2 seconds. Because of the above considerations, it is necessary to carry out the reaction at about 25-30 percent conversion per pass and at reduced butylene partial pressure in order to prevent butadiene degradation and excessive by-product formation. An additional problem is presented by the fact that the reaction is highly endothermic, requiring an external heat source of about 725 Btu/per pound of butenes converted.

One commercial solution of these problems employs steam as a heat conveying medium to provide sufficient heat capacity to reduce temperature drop through the reactor to a workable range, 50-75° F., and simultaneously lower butylene partial pressure to the desired range of 100 millimeters. Excessive butylene temperatures are avoided by superheating the steam diluent above the reaction temperature and mixing it with the hydrocarbon preheated to 100-200° F. below reaction temperature just before it contacts the catalyst. An important advantage is that steam reacts with coke to form water gas at the temperatures employed for dehydrogenating butylene, allowing regeneration of the catalyst by steam alone.

A catalyst stable and active in the presence of steam was a requisite for the steam-dilution process, dehydrogenation catalysts used in early development work not being satisfactory from this standpoint. Extensive laboratory and pilot-plant tests resulted in development of a suitable catalyst for this process, which also gave higher butadiene yields and lower by-product formation than previously known catalysts under similar conditions.

The dehydrogenation process as finally developed is indicated on the flow sheet, and utilizes two single fixed-bed reactors, with equal cycle times on reaction and regeneration. Butylene from storage is preheated by means of heat exchange and a furnace to not over 1100° F., while the diluent steam is superheated to some 1300° F., and jet-type mixers are employed for contacting the two streams directly over the reactor-catalyst bed. A 25-pound pressure drop is taken across the mixing nozzles to insure adequate mixing energy.

Reaction products are quenched to below 1000° F. as they leave the catalyst bed by means of a pumped stream of condensate. The hot reactor effluent is further cooled in a waste-heat boiler and a series of quench towers, a condensate containing the bulk of the C_4 hydrocarbons finally being charged to a fractionator for stabilization by separation of a light overhead stream and to a rerun tower where a polymer bottoms stream is produced and the crude butadiene is condensed as an overhead product in conjunction with other C_4 hydrocarbons, this mixture constituting the feed to a subsequent purification process step where commercial butadiene is produced by extraction with cuprous salt solutions, or by other means.

Non-condensable vapors from the cooled reactor effluent condensate separator are passed through an absorber for recovery of C_4 hydrocarbons. Oil from the reactor effluent quench system is in a closed circulation system, but is maintained in satisfactory condition by means of a small make-up feed stream and a lean-oil stripper which continuously eliminates a small bottoms stream to slop. Reactor effluent polymer from the rerun tower is also rejected to slop. Steam generated in the waste heat boiler is utilized as dilution steam feed to the steam superheater furnaces, in conjunction with high-pressure steam from outside sources. Water condensed from the reactor effluent in the water separator is stripped of any volatile hydrocarbon contaminants in a water-stripping tower and is then discharged to a water-blowdown system.

The above data and the accompanying flow diagram are available here through cooperation of Standard Oil Development Company, New York.

BUTADIENE PROCESS
COURTESY, STANDARD OIL DEVELOPMENT COMPANY
GAS TO FUEL
K.O DRUM
CHILLED WATER
DRY GAS
WATER
STABILIZER
ABSORBER
RECYCLE BUTADIENE FROM RUBBER PLANT
HOT
COLD
REFLUX DRUMS
STABILIZED C4 CUT
RERUN TOWER
WATER
LIVE STEAM
REFLUX DRUM
SPHERE
EXTRACTION SECTION FEED
POLYMER
PURGE OIL TANK
SLOP
FAT OIL
LEAN OIL
UNSTABIZD. C4 CUT
STEAM
CONDENSATE SEPARATOR
CHILLED WATER INTER.STG K.O DRUM
STEAM SUCTION K.O DRUM
LEAN OIL STRIPPER
2 STAGE COMPRESSOR
WATER TO BLOWDOWN SYSTEM
2 DEHYDROGENATION SECTION FEED SPHERES
WATER STRIPPER
WATER QUENCH TOWER
WATER SEPARATOR
OIL QUENCH TOWER
WASTE HEAT BOILER
4 STEAM FURNACES
I BUTENE FURNACE
REACTOR
H.P STEAM
CONDENSATE
MAKE UP LEAN OIL TANK

Butane Isomerization Process

THE M. W. KELLOGG COMPANY

THE isobutane requirements during the war for the alkylation of olefins was far greater than the available supply from natural or cracking sources. Catalytic processes were, therefore, developed to isomerize the more abundant normal butane to isobutane.

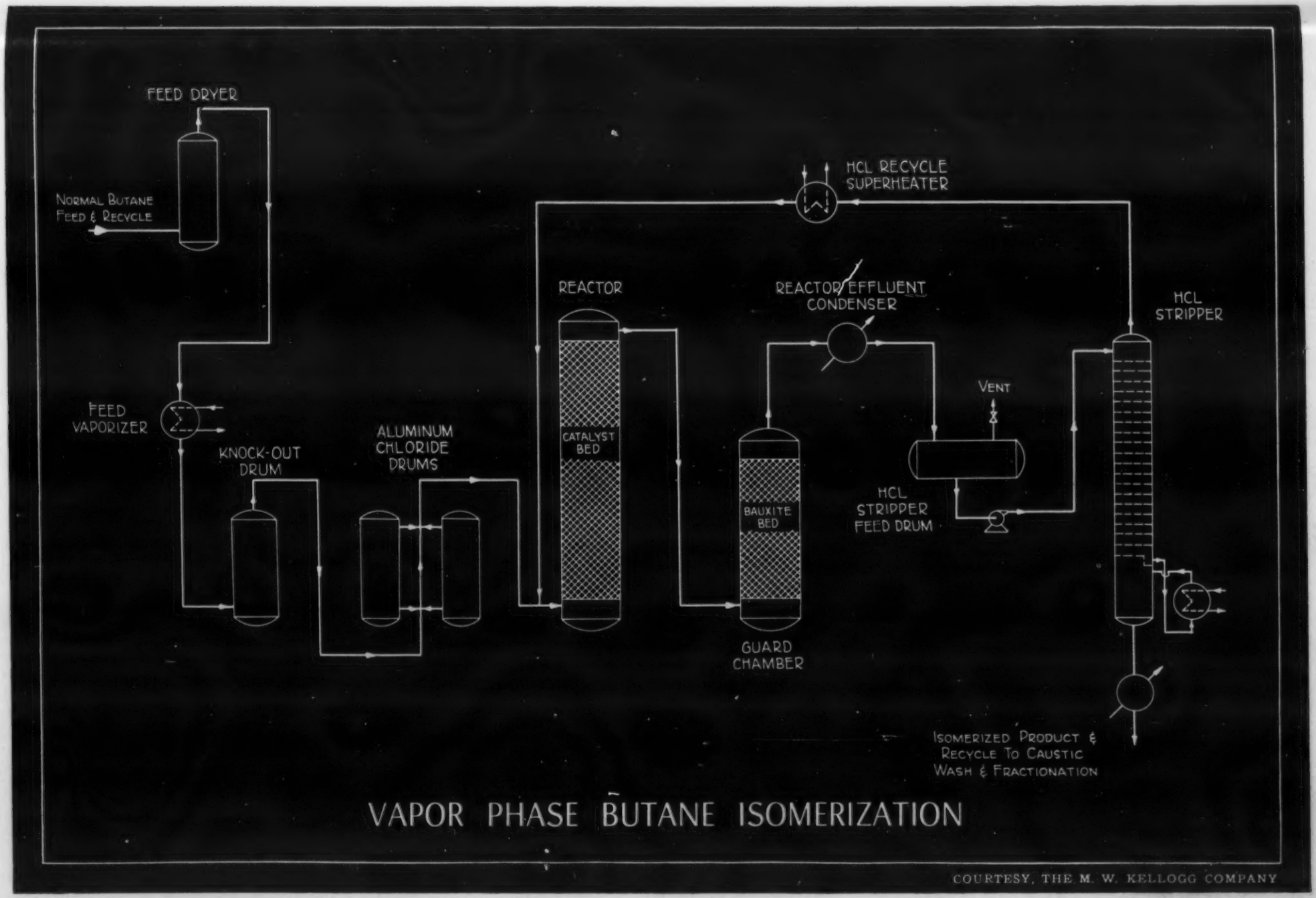
Practically all of the processes used aluminum chloride as the catalyst with the aid of some promoter. The process recommended by Kellogg uses aluminum chloride on a Porocel support as the catalyst and HCl as the promoter.

Normal butane mixed with 2 to 5 percent HCl is passed in vapor phase over a fixed bed of aluminum chloride on Porocel at 250° to 300° F. and approximately 250 psig. The pressure is set just below the minimum required to maintain the butane in the vapor phase. The effluent from the reactor goes to an HCl stripper from the top of which the HCl is recycled to the process. The bottoms from the stripper, after being washed with caustic or water, are sent to an isobutane tower to separate the isobutane product from the normal-butane recycle. If, as is usually the case, the isomerization unit is an integral part of an alkylation unit, the separation of isobutane from normal butane may be done in the isobutane tower of the alkylation unit.

Catalyst life varies from 200 to 400 gallons per pound of aluminum chloride. At intervals, as the activity of the catalyst drops, additional aluminum chloride is sublimed on to the Porocel bed by passing a small portion of the reactor feed through aluminum chloride drums. The Porocel bed usually lasts six to twelve months. HCl consumption is negligible.

The conversion per pass averages around 40 percent. A negligible amount of propane and lighter gases is produced in the reaction, so that the overall isobutane yield on recycling is approximately 98 percent on net butane feed.

Licenses for the butane isomerization process are available from The M. W. Kellogg Company.



VAPOR PHASE BUTANE ISOMERIZATION

COURTESY, THE M. W. KELLOGG COMPANY

Catalytic Alkylation Process

THE M. W. KELLOGG COMPANY

ALKYLATION as practiced by the petroleum industry is the catalytic method of combining an isoparaffin (usually isobutane) with an olefin (propylene, butylene or amylene) to produce saturated iso-compounds. The product of the reaction—alkylate—has a high-octane rating and is therefore employed as a blending agent in aviation and motor gasoline. It also has high heat value, giving it more power than other hydrocarbon fuels.

The catalyst used is either sulfuric acid or hydrofluoric acid. Yields and quality of products with either catalyst are essentially the same. The majority of the installations, however, employ the lower-cost sulfuric acid since it is less corrosive and the spent acid can be re-used in other refinery treating operations.

In the Kellogg flow sheet, the olefin is dissolved in concentrated sulphuric acid and violently agitated with an excess of isobutane at a temperature of 34-45° F. and a pressure several pounds above atmospheric. Under these conditions, the reaction proceeds readily.

Since the reaction is highly exothermic, the heat of reaction is removed by auto-refrigeration. Conditions are maintained at the bubble point of the mixture and a sufficient amount of hydrocarbon is allowed to vaporize to cool the mixture to the control temperature. The vaporized hydrocarbon is compressed, condensed and returned to the system.

The product of the reaction is separated from acid in the center section of the combination reactor-settler and after caustic treatment is pumped to the isobutane tower.

The isobutane overhead from this tower is returned to the process while the alkylate bottoms containing any normal butane present in the original feed is sent to a debutanizer tower where butane is removed as an overhead product. The alkylate bottoms usually are separated in a rerun tower into a light aviation cut and a heavy alkylate.

The major factors affecting product quality and other operating results are:

1. Reaction Temperature
2. Olefin Concentration
3. Isobutane Concentration
4. Acid Strength

Each of these factors will be discussed briefly.

1. Reaction Temperature

The lower the temperature at which the reaction is carried out, the better the quality of the alkylate and the lower the acid consumption. On the other hand, below 35° F. the sulfuric acid-hydrocarbon emulsion tends to get so stiff as to make operations difficult.

2. Olefin Concentration

The lower the concentration of olefin is kept at any time, the lower is the tendency toward polymerization and the higher the tendency toward alkylation. This is effected by circulating large volumes of emulsion as compared with the amount of material in the reactor, and controlling the addition of olefins so as to maintain this low concentration.

3. Isobutane Concentration

Normal butane does not enter into the reaction and is a harmful diluent in the sense that it reduces the chances for contact between the isobutane and the olefin-bearing acid. It is, therefore, advisable to keep the isobutane concentration in the reactor as high as possible.

4. Acid Strength

While 98 percent is the strength of the acid usually added to the process, a more important factor is the average strength of the acid contained in the reactor. In the case of butylenes and propylenes, this strength should not be less than 90 percent. In the case of amylenes, the acid strength can drop to 85 percent without seriously affecting the quality of the alkylate.

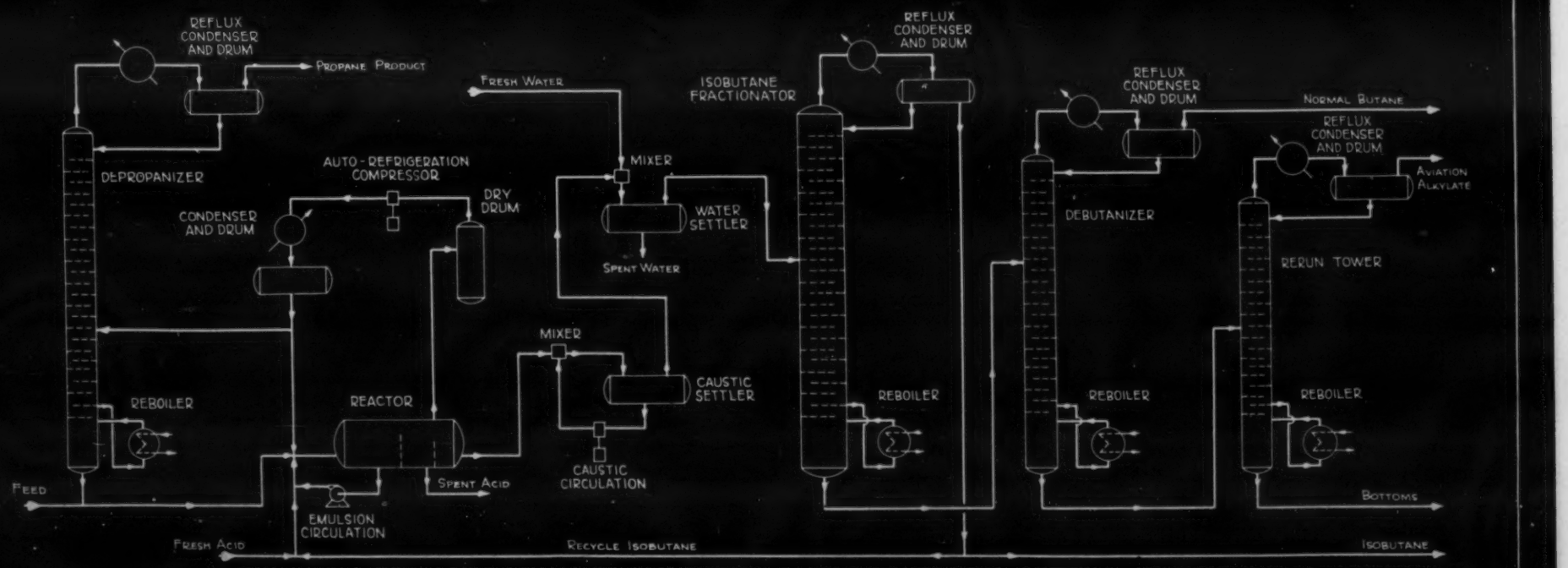
Most of the alkylation units in this country were designed for operation on butylenes. This olefin gives the best product quality and the lowest acid consumption. The same units, however, also can be used for alkylating propylenes and amylenes. Typical results are given in the following table.

Typical H_2SO_4 Alkylation Results

	Butylenes	Propylene	Amylenes
Alkylate—Volume Percent on Olefin	170	170	160
Isobutane—Volume Percent on Olefin	110	125	110-120
Acid Consumption—Lb. per gal. Alkylate	0.7	3	1-2
ASTM Octane rating of Alkylate	94	89	90
Reid Vapor Pressure of Alkylate—lb./sq. in.	3-4	5	2
End Point of Alkylate—°F.	350	350	350

Because the sulfuric acid is of high concentration and the plants are operated at below atmospheric temperature, corrosion is negligible and steel equipment can be used throughout. Individual requirements vary widely and the economics depend largely on the availability of the raw materials at low cost.

The M. W. Kellogg Company, New York, has been appointed licensing agent for the process for the United States and foreign countries. Licenses granted are under the patents and patent rights of Texaco Development Corporation, Standard Oil Development Company, Shell Development Company, and Anglo-Iranian Oil Company, Ltd.



SULFURIC ACID CATALYTIC ALKYLATION

COURTESY, THE M. W. KELLOGG COMPANY

Catalytic Cracking Process

UNIVERSAL OIL PRODUCTS COMPANY

THE fluid-catalytic-cracking process is adaptable to the needs of large and small refiners. Units designed by Universal Oil Products Company, in capacities ranging from 2600 to 18,000 barrels a day, have been in successful operation for years. UOP improved fluid-catalytic-cracking units can be built and operated at costs comparing favorably with the cost of thermal-cracking units of comparable capacity.

Development and commercialization of the fluid catalytic cracking process marks an important forward step in petroleum refining. It produces gasoline of a different character from that produced by thermal cracking.

The process is flexible both as to charging stock and products. It can successfully crack kerosine, light or heavy gasoil and some types of reduced crude. It can be operated to produce either aviation gasoline, high-grade motor gasoline or raw materials for the production of chemicals and synthetic rubber.

Principal modifications in the improved unit as compared to the units of earlier design are:

1. The reactor and regenerator have been combined in a single tower which is erected as a self-supported column and completely shop-fabricated for moderate-sized units. The heavy structure required in the earlier units has been eliminated.

2. The large regenerator riser has been eliminated, and the regenerator standpipe has been considerably shortened. There has been a general reduction and simplification of small piping.

3. Efficiency of both the reactor and regenerator has been improved by the use of additional distribution grids in each section.

4. The air supplied for combustion passes through the regenerator countercurrent to the catalyst. This readily permits multistage stripping and total utilization of oxygen.

The oil feed is charged as a liquid at the base of the reactor riser, while hot catalyst from the regenerator enters the riser at the point of oil injection, supplies the process-heat requirements and is transported upward by the vaporized oil into the reactor. Spent catalyst is continuously withdrawn from the upper part of the reactor dense-phase to hold a level in the reactor, and passes downward through the stripping section into the regenerator. Three zones are available for stripping in this design. The stripping medium is injected at the bottom of both the internal and the external strippers. Additional stripping of the spent catalyst is obtained in the upper section of the regenerator where the bulk of the flue gas from the combustion of coke in the lower sections contacts the incoming catalyst. The catalyst passes downward through the regenerator countercurrent to the air supplied at the bottom for regeneration.

Regenerated catalyst flows from the lower section of the regenerator to the reactor riser at a rate controlled to maintain a constant reactor temperature. The regenerator flue gas is vented to the atmosphere through an internally mounted cyclone separator which recovers entrained catalyst. The hydrocarbon products from the reactor pass to the fractionator likewise through an internally mounted cyclone separator which recovers entrained catalyst and returns it to the catalyst bed.

By building the regenerator below the reactor it is now possible to pass the air for regeneration countercurrent to the flow of catalyst. With the countercurrent flow and staging of the regenerator with grid plates the oxygen content of the outlet gas can be reduced to ½ to 1 percent while in previous units two to three times as much unused oxygen normally escapes from the regenerator. This feature of the new design was not possible in the older units.

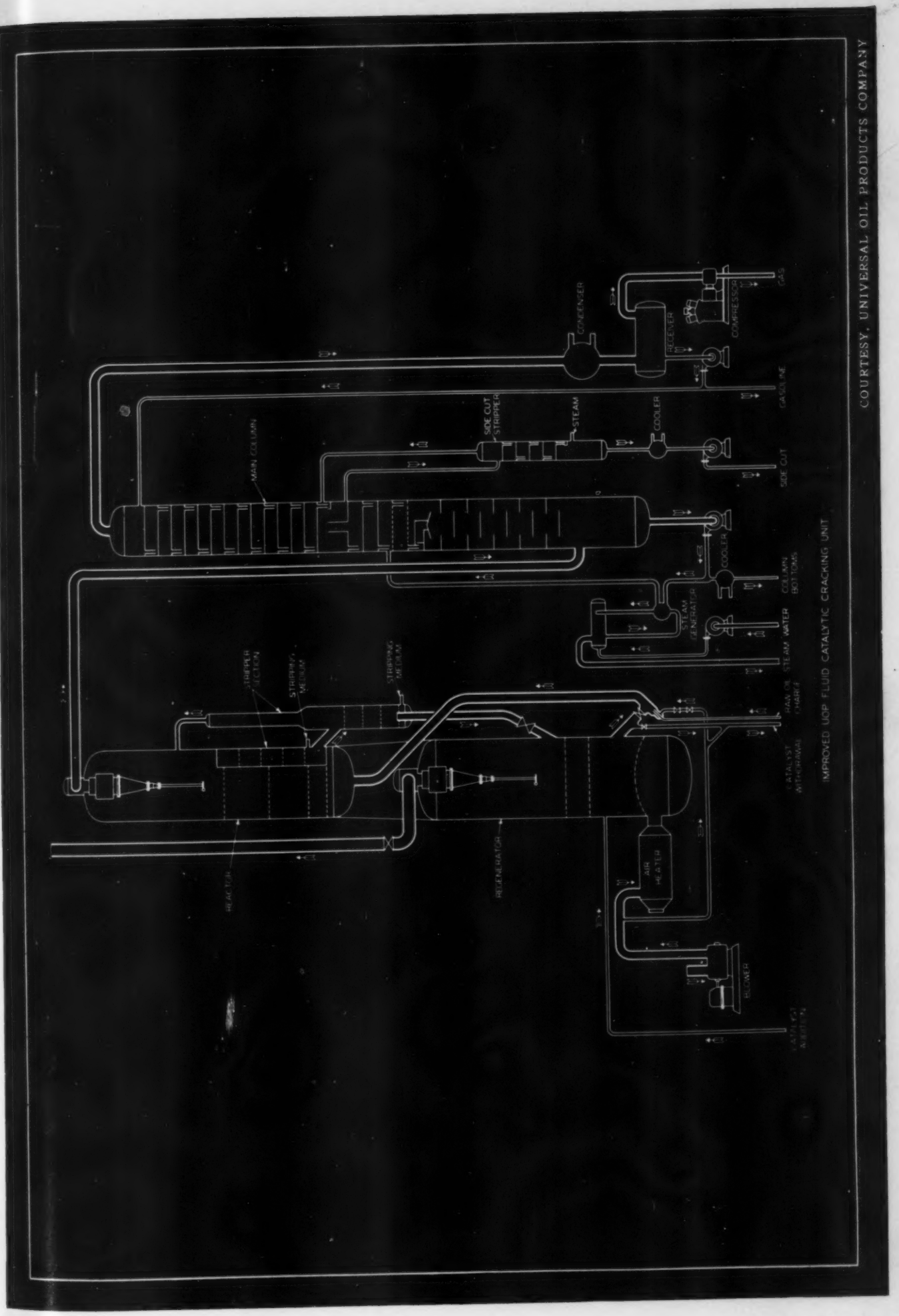
The latest type unit can utilize either synthetic or natural catalyst. In its most simple design synthetic catalyst which has a microspherical form is used. Because of the spherical shape of the particle and its greater strength as compared with the ground powdered catalysts used during the war, this catalyst has very low erosion and attrition rates. In addition, the M.S. catalyst is much more uniform in particle size than the ground catalyst. With the reduced formation of fines by attrition, plants using M.S. catalyst can dispense with secondary catalyst recovery while still maintaining a low catalyst loss.

Economic consideration of market requirements and operating costs will govern the choice of catalyst. The silica- alumina catalyst gives the highest octane number but a lower gasoline yield, while somewhat more gasoline having a lower octane number is produced from the silica-magnesia catalyst. The natural catalyst gives a gasoline yield and octane rating intermediate to the values obtained by the two synthetic catalysts.

The accompanying table shows the yields obtained in pilot-plant once-through and recycle cracking of 31° API Mid-Continent gasoil over silica magnesia catalyst.

Fluid Catalytic Cracking 31° API Mid-Continent Gasoil (Silica-Magnesia Catalyst) Pilot-Plant Results

OPERATION	Once-Through	Recycle
Conversion, percent	65	80
Yields, Volume, percent:		
Debutanized gasoline	53.0	66.0
Cycle oil	35.0	20.0
Butylenes	6.0	6.7
Butanes	6.0	5.3
Propylene	3.8	4.1
Propane	1.8	1.8
C_2 and lighter gas, weight percent	2.3	2.7
10 lb. RVP catalytic gasoline	55.9	69.5
10 lb RVP polymer gasoline	6.6	7.4
Total 10 lb. RVP gasoline	62.5	76.9
Octane Numbers of 10 lb. Catalytic Gasoline:		
Motor method clear (F-2)	79.8	79.1
+ 3 cc TEL/Gal.	86.1	85.0
Research method clear (F-1)	89.6	90.2
+ 3 cc TEL/Gal.	96.2	96.4


IMPROVED UOP FLUID CATALYTIC CRACKING UNIT
COURTESY, UNIVERSAL OIL PRODUCTS COMPANY
CONDENSER
RECEIVER
COMPRESSOR
GAS
GASOLINE
SIDE CUT STRIPPER
STEAM
COOLER
SIDE CUT
MAIN COLUMN
COOLER
COLUMN BOTTOMS
STEAM GENERATOR
STEAM
WATER
STRIPPER SECTION
STRIPPER MEDIUM
STRIPPING MEDIUM
CATALYST WITHDRAWAL
HOT CATALYST CHARGE
REACTOR
REGENERATOR
AIR HEATER
BLOWER
AIR TO REGENERATOR

Catalytic Desulfurization Process

UNIVERSAL OIL PRODUCTS COMPANY

DESULFURIZATION of petroleum distillates is becoming of greater importance as the use of high-sulfur crudes becomes more widespread. Sulfur reduction of gasoline is of great interest because of the improved octane number and response to tetraethyl lead that can be effected thereby.

Certain inexpensive naturally occurring adsorptive clays exert a selective catalytic activity toward decomposition of the sulfur compounds found in petroleum distillates at a temperature below which cracking of the hydrocarbons takes place. This phenomenon is the basis of a desulfurization process developed by Gray Process Corporation. The process is simple as to equipment, easy of operation and gives excellent results.

This process, licensed by Universal Oil Products Company, finds its widest application in desulfurizing straight-run distillates.

The stock to be desulfurized is charged through heat exchangers to a heater where it is vaporized and heated to about 700° F. The heated vapors pass directly to the catalyst tower wherein the sulfur compounds are decomposed to hydrogen sulfide. Effluent vapors from the catalyst tower pass through the charge heat exchanger and are then condensed. Hydrogen sulfide is removed by any of the conventional methods and the desulfurized product sent to storage.

If straight-run gasoline is treated, rerunning the product is not necessary, unless it is desired to recover a light and a heavy fraction for blending purposes.

If stabilization of the raw straight-run gasoline is required for light-ends removal, the stabilizer can be located after the desulfurizing unit, in which case hydrogen sulfide will be removed with the stabilizer gases.

Sulfur reduction depends upon catalyst activity (being greater for fresh clay) temperature of catalyst bed, distillation range of stock and type of sulfur compounds present. For most straight-run gasolines, extent of desulfurization will range from 60 to 95 percent of the total sulfur in the charge.

Octane number improvement over the raw gasoline ranges from 1 to 3 numbers without tetraethyl lead and may be as high as 12 numbers on the gasoline at the 3 cc tetraethyl lead level, depending on sulfur content and boiling range of the material treated. Some isomerization of straight- to branched-chain hydrocarbon seems to occur on certain stocks as the extent of sulfur removal cannot always account for the octane number improvements obtained. It is, therefore, pertinent to examine stocks that do not seem to merit consideration from a sulfur standpoint alone. On some high-sulfur straight-run stocks, this process may compare favorably with reforming from a leaded octane standpoint.

Since the process decomposes organic sulfur compounds to hydrogen sulfide and a hydrocarbon molecule, with only the hydrogen sulfide being removed, volumetric losses are extremely low—usually in the range of 0.5 to 1 percent.

Operating costs exclusive of amortization will vary from 6 to 15 cents per barrel treated, depending upon local utility values, amount of hydrogen sulfide to be removed, and method of hydrogen sulfide removal. Clay life when treating straight-run stocks ranges from 5,000 to 30,000 or more barrels per ton. This long life keeps the cost of clay so low that regeneration is not economical and is seldom practiced.

HIGH TEMPERATURE CLAY DESULFURIZATION UNIT
COURTESY, UNIVERSAL OIL PRODUCTS COMPANY
CHARGE
CHARGE PUMP
HEATER
GAS
CATALYST TOWER
EXCHANGER
CONDENSER COOLER
CAUSTIC CIRCULATING PUMP
MIXER
CAUSTIC SETTLER
CAUSTIC TO AND FROM STORAGE
TREATED PRODUCT TO STORAGE

Catalytic Polymerization Process

THE M. W. KELLOGG COMPANY

CATALYTIC polymerization is a process for chemically uniting the propylenes and butylenes produced by thermal or catalytic cracking to produce a liquid which is an excellent component of motor and premium gasoline, and, after hydrogenation, of aviation gasoline.

Under normal circumstances the average refiner is able to include an appreciable amount of butanes in motor gasoline, but he usually has excess C_4's over and above this amount. Consequently, it is usually economic to polymerize propylene and butylene, and merely use the remaining saturated butanes for Reid-vapor-pressure fortification. Such catalytic polymerization moves propylenes and butylenes from fuel gas into the hydrocarbon group boiling in the gasoline range.

The catalyst employed in Kellogg units is copper pyrophosphate. It has a normal life of 100-150 gallons of polymer per pound of catalyst.

The liquid stream of C_3 and C_4 hydrocarbon reactants from the gas-recovery system is first treated to remove hydrogen sulfide and then washed with caustic to remove mercaptan sulfur. It is then heated to 300-400° F. and introduced into the reactor which is kept at about 900 psig to maintain the material in the dense phase.

After the reaction occurs, the reactor effluent proceeds to conventional stabilizing equipment as follows:

The hot product gases leaving the reactor exchange a portion of their heat to the incoming charge and then pass to a depropanizer where propane and any unconverted propylene is routed either to plant fuel or to bottled gas. The depropanized product flows to a debutanizer where butanes are removed, leaving the high-octane polymer gasoline as the bottom product. The butanes produced are used for motor-fuel-blending purposes.

Essentially there are two types of operation, depending on the feed stock.

(a) *Codimer Operation,* which is polymerization of butylenes only and which uses a hydrocarbon feed stock of essentially 100 percent C_4's. The operation usually is carried out at temperatures on the order of 300° F. The product, called codimer, is unsaturated, but when hydrogenated has a clear ASTM octane number on the order of 95 and is, therefore, a very acceptable component for aviation gasoline.

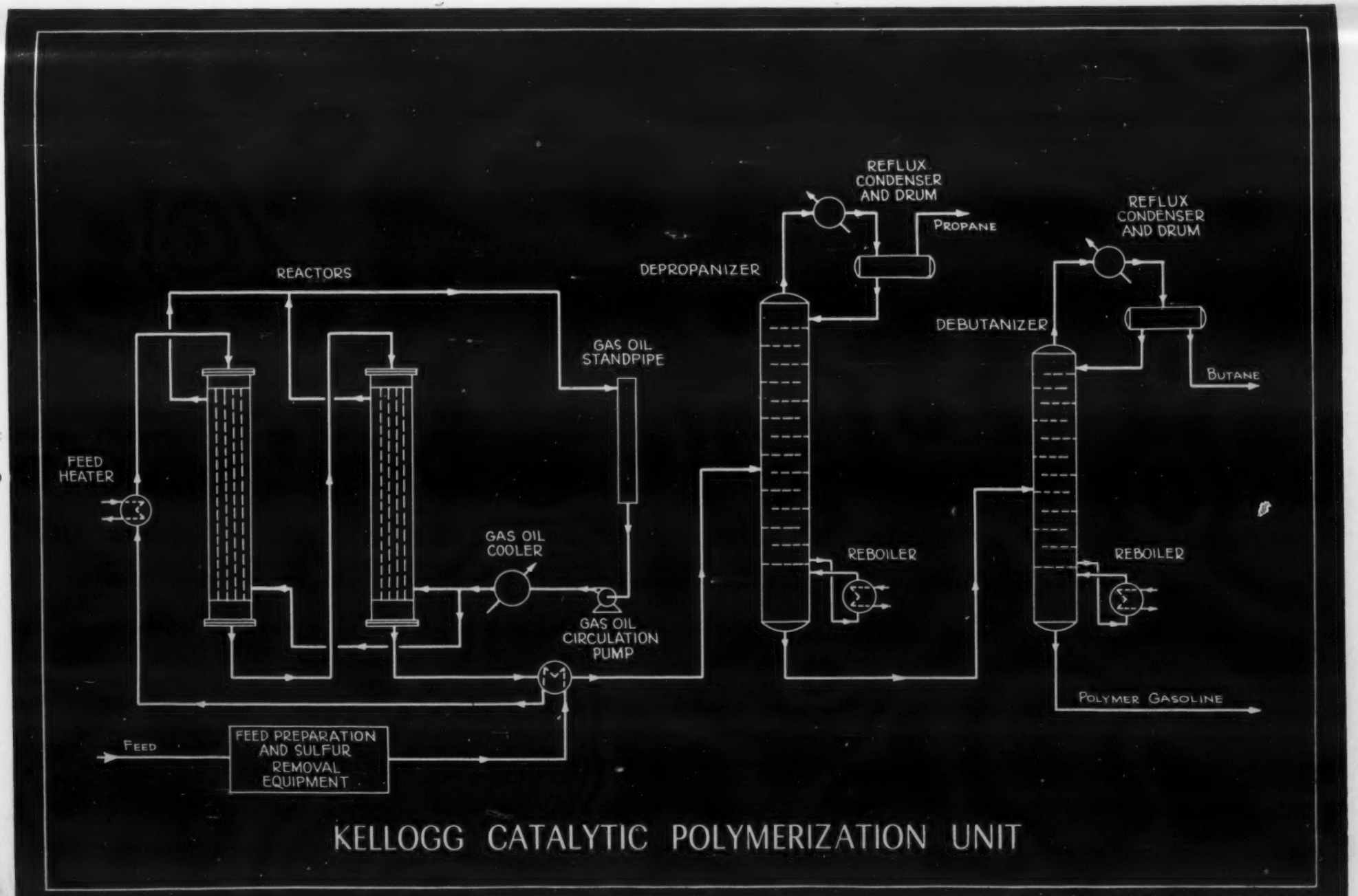
(b) *Motor Fuel Operation,* uses for a feed stock mixtures of C_3's and C_4's and the operation usually is carried out at approximately 375-400° F. The polymer is unsaturated and has a clear octane of about 83 ASTM and 96-98 CFRR. Its blending value, however, is considerably over 100 by either rating method when the percentage in the blend is not more than 10 or 15 percent.

A common type of reactor is the tubular reactor which is essentially a heat exchanger in which the tubes are filled with catalyst and surrounded by a bath of gasoil which is circulated to remove the heat of reaction. The reaction temperature is automatically controlled.

Typical Catalytic Polymerization Results

	Codimer Operation	Motor Fuel Operation
Conversion, percent of Olefin	80	90
Octane No.—ASTM Clear	95	83
Octane No.—CFRR Clear	....	96-98
RVP of Polymer, psi	2.0	2.0
IBP of Polymer, °F	148	170
EP of Polymer, °F	421	431
Gravity of Polymer, °API	60.4	60.1

Licenses are available from The M. W. Kellogg Company. Licenses are granted under the Polymerization Process Corporation patents, and patent rights of Phillips Petroleum Company, Standard Oil Company (Indiana), Standard Oil Company (New Jersey), The Texas Company, and The M. W. Kellogg Company.



KELLOGG CATALYTIC POLYMERIZATION UNIT

COURTESY, THE M. W. KELLOGG COMPANY

Chlorex Solvent Refining Process

CARBIDE AND CARBON CHEMICALS CORPORATION

CHLOREX (B. B′-Dichlorethyl ether) is a colorless liquid of ethereal and not unpleasant odor. Aside from the desirable property of high selectivity, this solvent is particularly well adapted to plant-scale usage. High specific gravity permits rapid separation of the two phases formed with oils. Its boiling point is well below the vaporization temperature of motor oils, and its low vapor pressure precludes the possibility of significant losses in storage. The freezing point of the material is so low that precautions are unnecessary. Since extraction with Chlorex is ordinarily carried out within the temperature range of 50° to 125°F. its low viscosity at these temperatures is a factor of importance. The flash point of the solvent is well above that considered hazardous. It is noninflammable at ordinary temperatures. It has been employed since 1933 on plant-scale.

PROPERTIES OF DICHLORETHYL ETHER

Structural formula	$Cl-CH_2CH_2-O-CH_2-CH_2-Cl$
Boiling point (sea level), °F. (°C.)	352.4 (178)
Freezing point, °F. (°C.)	—61 (—51.7)
Specific gravity at 20°/20°C	1.222
Viscosity, centipoises at 25.5°C.	2.0653
Viscosity, S U at 77°F. (25°C.) sec.	32
Viscosity, S U at 32°F. (0°C.) sec.	39
Vapor pressure at 100°F. (37.8°C.) mm.	2
Latent heat of vaporization B.t.u./lb. (cal./gram)	115.4 (64.1)
Specific heat at 85°F. (29.4°C.)	0.369
Flash (closed cup), °F. (°C.)	168 (75.6)
Solubility in water at 20°C. %	1.01
Solubility in water at 90° C. %	1.71

The application of Chlorex to commercial production can be carried out with a minimum of investment. Optimum extraction temperatures are not greatly different from atmospheric temperatures, so investment and operating cost for control of extraction temperature is low.

Prior to the construction of plants a detailed laboratory investigation of the possibilities of the process was carried out. This indicated that motor oils far superior to those on the market could be made at a moderate cost. As a result the process was established on a commercial basis.

On the opposite page appears a flow diagram of a Chlorex plant using conventional design of extraction equipment and vacuum distillation for continuous recovery of the solvent from the raffinate and extract solutions. Operation is as follows:

The charge oil is pumped through a heat exchanger M, operated either as a cooler or heater, depending on whether water or steam is circulated through the unit. Chlorex-extraction temperatures range from 60 to 125° F., depending on the stock and the results desired. The rate of pumping the charge oil is under automatic flow control. The preheated or pre-cooled oil is mixed in the first-stage pipe mixer, with the extract solution (thrice-used Chlorex) from the second stage. This mixture settles in the first-stage settling tank. The extract solution from the first-stage settler is pumped, under automatic float control, to the extract-solution distillation unit for continuous recovery of the solvent. The raffinate solution from the first stage flows under first-stage pressure, and is mixed in the second-stage pipe mixer with extract solution from the third stage. This mixture settles in the second-stage settling tank. Raffinate solution from the second-stage settler is mixed with extract solution from the fourth stage (once-used Chlorex) in the fourth-stage pipe mixer. This mixture settles in the third-stage settling tank. The raffinate solution from the third-stage settling tank is mixed with fresh Chlorex in the fourth-stage pipe mixer. This mixture settles in the fourth-stage settling tank. The final raffinate solution, from the fourth-stage, is pumped under automatic control to the raffinate-solution distillation unit for continuous recovery of the solvent. When using more stages, the operation of the extraction equipment is the same as described above.

A continuous countercurrent extraction tower for contacting Chlorex and oil was installed in one Chlorex plant in the fall of 1936. This tower, 5′ 6″ in diameter and 67′ high, has performed efficiently with neutral charging rates up to 1000 barrels per day, and steam refined cylinder stock at a rate of 600 barrels per day with oil as the continuous phase and Chlorex as the dispersed phase. Oils of high pour point can be treated with Chlorex in towers of this type without mechanical difficulty of any kind by proper control of the temperature in the tower and the temperature of the extract solution leaving the system. Important advantages of the countercurrent tower over a system of settlers and mixers, which have been confirmed by this installation, include lower solvent holdup in the extraction system, high stage equivalence and thus higher yields of oil of given quality, simplification of operation, lower investment and less space requirements.

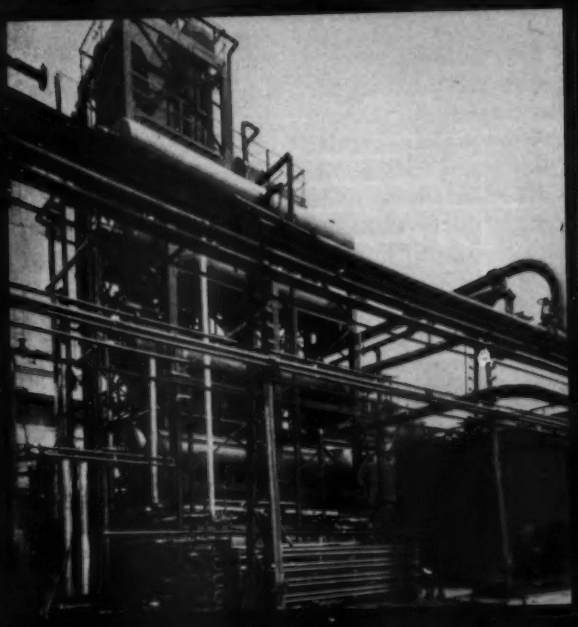
The solvent-recovery system is maintained under a vacuum of 26 inches to 28 inches mercury, and the solvent is completely recovered from the oil at a maximum stripping temperature of 300 to 325°F. The distillation equipment shown is steam-heated. Direct-fired vacuum-distillation units are also being used for recovering Chlorex, and the choice of equipment depends to a large degree on whether sufficient live steam is available to operate the unit.

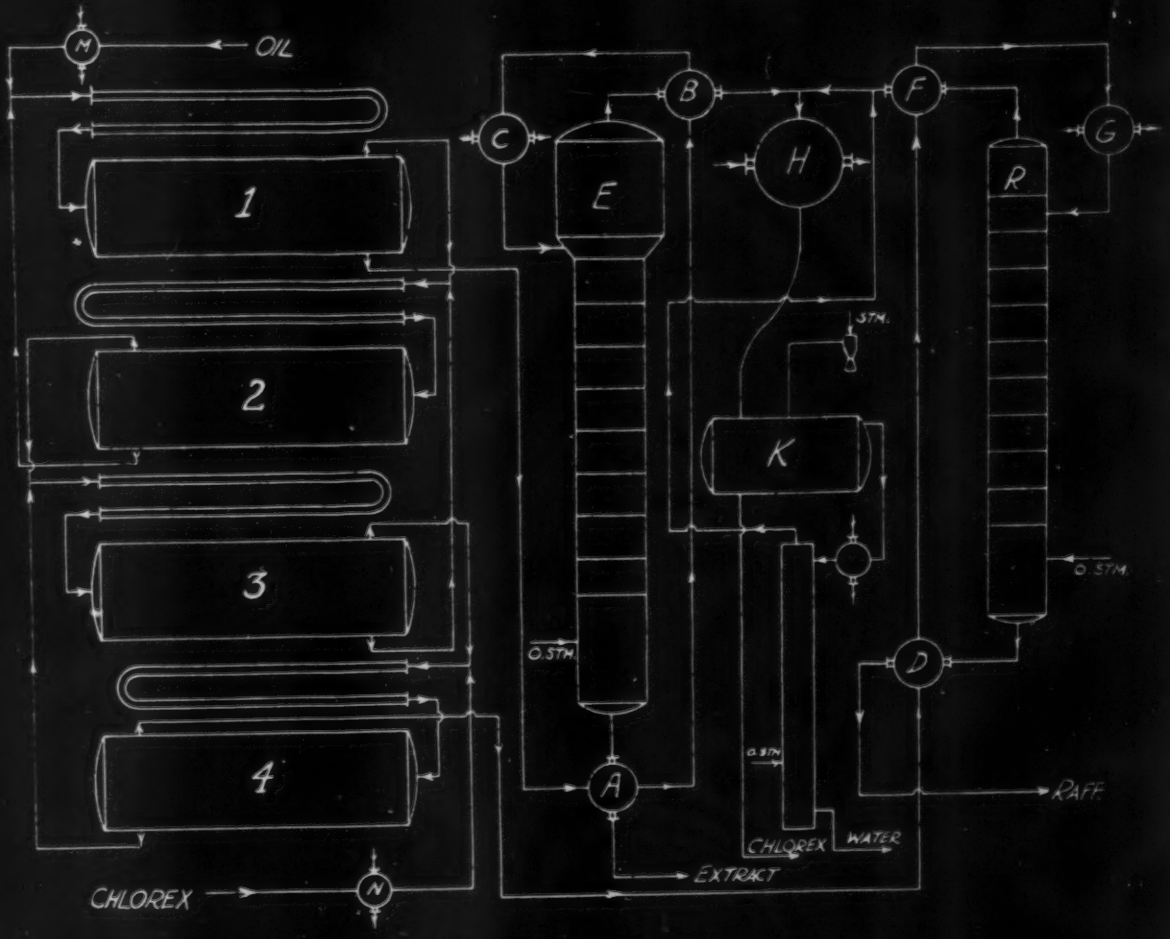
The final extract solution from the first-stage settling tank passes through a heat exchanger, A, utilizing the heat in the stripped bottoms from the extract-distillation unit, E. Further heat is supplied in a second heat exchanger, B, utilizing the heat in the hot Chlorex water vapors from the extract unit. Any additional heat required is supplied with a steam pre-heater, C. The extract solution enters the stripping column near the top. Open steam is applied at the bottom of the column. Re-boiler coils may be used to maintain the temperature throughout the column, which is a simple fractionating column with bubble trays.

The final raffinate solution from the fourth stage is heated with bottoms and vapor heat exchangers, D and F, and a steam pre-heater G. As the raffinate solution will contain only 15 to 25 percent solvent, the raffinate-stripping unit, R, is smaller than the extract unit, E, where the bulk of the solvent is recovered. Hot Chlorex water vapors from the extract and raffinate-solution distillation units pass through a water-cooled condenser, H—the condensed Chlorex and water flowing to a settling tank, K, where the solvent is separated from the water by gravity and returned to storage. The water will be saturated with Chlorex with a solvent content of approximately 1 percent. This water is fed through a preheater to a small, packed column, which may be readily constructed from a piece of casing. Open steam is added near the bottom. Water which is completely denuded of Chlorex flows to the sewer under automatic float control from the bottom of this tower, while the vapors which are rich in Chlorex pass to the raffinate-extract vapor condenser, H. A vacuum is maintained on the system by means of a steam-jet vacuum pump. It is not necessary to operate the water-stripping column under a vacuum. A Chlorex plant of this design, for treating 1000 barrels of oil per day, will require a total ground space not over 50 feet square. From 15,000 gallons to 20,000 gallons of solvent is adequate to place such a plant in operation.

Operating costs in plants with continuous vacuum-distillation equipment will run from 0.4 to 0.5 cents per gallon of oil charged, which includes steam, water, electricity, fuel, Chlorex loss, maintenance, depreciation, and labor. In five Chlorex plants using vacuum distillation for recovering the solvent over-all losses have ranged from .04 to .08 percent of the total volume of Chlorex circulated.

Chlorex is sold in tank cars. No royalty is added for the use of the process. Chlorex is sold by Carbide and Carbon Chemicals Corporation, 30 East 42nd St., New York, N. Y.





CHLOREX EXTRACTION PROCESS

COURTESY, CARBIDE AND CARBON CHEMICALS CORPORATION

Cobalt Molybdate Desulfurization Process

UNION OIL COMPANY OF CALIFORNIA

THE depletion of low-sulfur-content crudes in the United States is forcing refiners to employ increasing amounts of high-sulfur-content, low-quality crudes of which there are considerable reserves. To date, the utilization of these latter stocks has been largely in the form of blends with the sweet crudes normally used in refinery operations. The sulfur-containing distillates have been handled by a variety of methods ranging from mercaptan removal only to mild catalytic reforming. However as the amount of high-sulfur stocks processed increases, the proportion of refractory thiophenes and thiophanes, largely unaffected by present treating processes, also is increased, and a definite need is arising for a more effective means of desulfurization.

The Cobalt Molybdate Desulfurization Process has been developed as an answer to the limitations of existing procedures. It represents a versatile method of sulfur removal capable of handling all types of petroleum distillate stocks encountered in normal refining operations, and is capable of removing sulfur from even the most refractory compounds associated with these stocks. Fundamentally, the process involves the conversion of organically combined sulfur to hydrogen sulfide by contact with a catalyst containing cobalt molybdate in the presence of hydrogen. Cracked gasoline stocks containing 3 percent by weight or more of sulfur have been treated to give products of as low as 0.01 percent by weight of sulfur in yields of 99-100 volume-percent. Heavy gas-oil stocks containing as much as 5.0 percent by weight of sulfur have been reduced in sulfur content to 0.5 percent or even lower, and with high yields. The accompanying diagram presents a simplified process flow for a unit to handle coke-still distillate of about 4.0 percent sulfur content and containing approximately 30 percent of material distilling in the lubricating-oil range. Referring to the diagram, the feed stock and hydrogen are preheated by exchange against the hot reaction products. The exothermic heat of reaction is of sufficient magnitude to make the process independent of the preheat furnace except during start-up periods. The catalytic treatment may be conducted over a wide range of temperatures, but for maximum yield and desulfurization, a temperature in the neighborhood of 800° F. is employed. The reaction products after exchange with the feed are cooled and passed to a separator. The liquid product from the separator may be caustic-washed to remove traces of dissolved hydrogen sulfide and then sent to storage. The gas from the separator, containing hydrogen, hydrogen sulfide and small amounts of hydrocarbons, is treated in an oil absorption unit at the reaction pressure and the separated hydrogen recycled to the reactors.

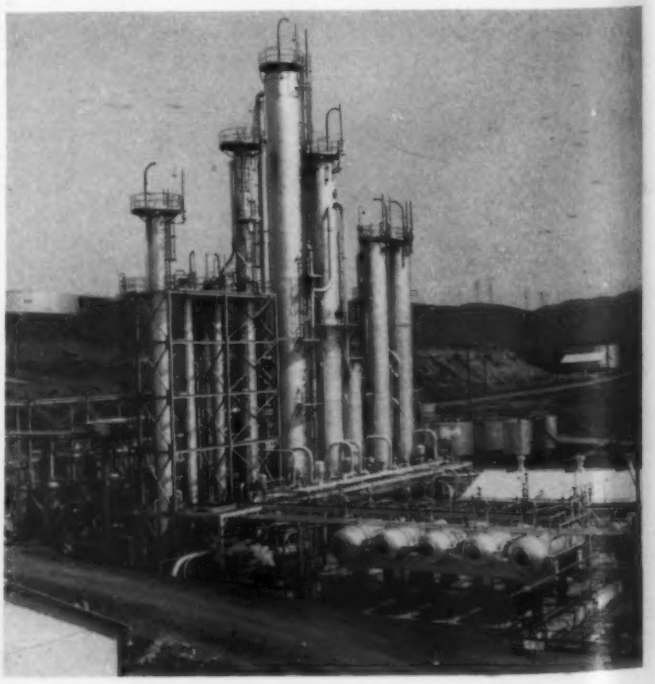
For the treatment of coke-still distillate, a multiple reactor construction is employed, three reactors being on stream while the fourth is being regenerated. Regeneration of the catalyst is accomplished in the conventional manner by adding air to a stream of inert combustion gas circulated through the catalyst bed. The characteristics of a typical coke-still distillate and the products resulting from its treatment by the Cobalt Molybdate Process are given in the following tabulation:

Characteristics of Feed and Products from Desulfurization of Coke Still Distillate

	Feed, Heavy Coker Gas Oil	Gasoline Fraction of Product	Gas Oil Fraction of Product
Yield, percent by Vol. based on Feed	....	8.4	91.1
Gravity, °API at 60° F.	19.4	47.7	28.1
ASTM Distillation Temp., °F.:			
Initial	482	164	480
10 percent	584	238	498
50 percent	699	320	612
90 percent	....	372	....
Maximum	760+	392	760+
Rec. percent	72.5	98.0	91.0
Sulfur Content, percent by weight	4.2	0.1	0.5
Acid Solubility, percent by Vol.	100.0	37.5	50.0
Knock Rating, CFR Motor Method:			
Clear	....	64.5	....
3.0 ml. TEL/gal	....	76.5	....
Viscosity, SUS at 100° F.	84	....	55
Pour Point, °F	65	....	45

The above is illustrative of only one type of application of the Cobalt Molybdate Process. Cobalt Molybdate treatment is unusually flexible and, if desired, can be used not only to remove sulfur compounds but simultaneously to improve the octane rating of gasoline stocks. In all cases the gasoline products have excellent lead susceptibilities. The products heavier than gasoline make good reforming and cracking stocks.

Detailed information on specific problems can be obtained from Union Oil Company of California, Los Angeles.



COBALT MOLYBDATE DESULFURIZATION PROCESS
REACTOR FURNACE
INERT GAS GENERATOR
REACTORS
GAS VENT
FUEL GAS
AIR
FEED STOCK
HYDROGEN
GAS SEPARATOR
CAUSTIC SCRUBBER
WATER WASHER
OIL ABSORBER
STRIPPER
H_2S
DESULFURIZED PRODUCT
CAUSTIC
WATER
COURTESY, UNION OIL COMPANY OF CALIFORNIA

Continuous Contact Filtration

FILTROL CORPORATION

CONTACT filtration is a process wherein petroleum lubricating oils are rapidly and efficiently refined by the simple procedure of mixing oil directly with Filtrol, the activated adsorbent made by Filtrol Corporation, heating for a short period and then separating the decolorized oil from the spent cake.

The superiority of this process over other methods of refining has been increased by recent improvements in equipment design and in bleaching adsorbents, which in turn have simplified the operation, improved product quality and lowered costs. Some of the major advantages which are now recognized are briefly summarized below:

Continuity of Operation. The contact process as now developed permits continuous operation from start to finish. This advance has been accomplished largely as a result of recent improvements in equipment manufacture.

Large Throughput with Minimum Space and Equipment Requirements. Inasmuch as this process provides ready adjustment of contacting temperature it is obviously possible to handle stocks of a wide viscosity range with but little effect on plant throughput. The cost of modern contact-plant installations is relatively low. Such a plant will occupy a space not greater than 80 feet wide by 120 feet long.

Ease of Switching from One Type Stock to Another. Due to the flexibility of the contact equipment and its small volume, changes from one type oil to another can be effected with minimum time loss.

Low Labor Cost. A modern 2000-barrel-a-day contact plant can be operated with two men per shift. Equipment design has undergone improvement to the extent that substantially each unit of the plant can be controlled automatically.

Rapid and Accurate Cost Analysis. Simplicity of the contact process makes it possible to obtain accurate daily cost data. Thus the management is constantly provided with true costs of the finished oils, either individually or collectively.

High Quality Products. Uniform and predictable colors, superior casts, lower carbon residues, improved emulsion and oxidation tests, are among the outstanding merits of this process. The use of fresh and uniformly standardized adsorbent in the contact process eliminates the uncertainties.

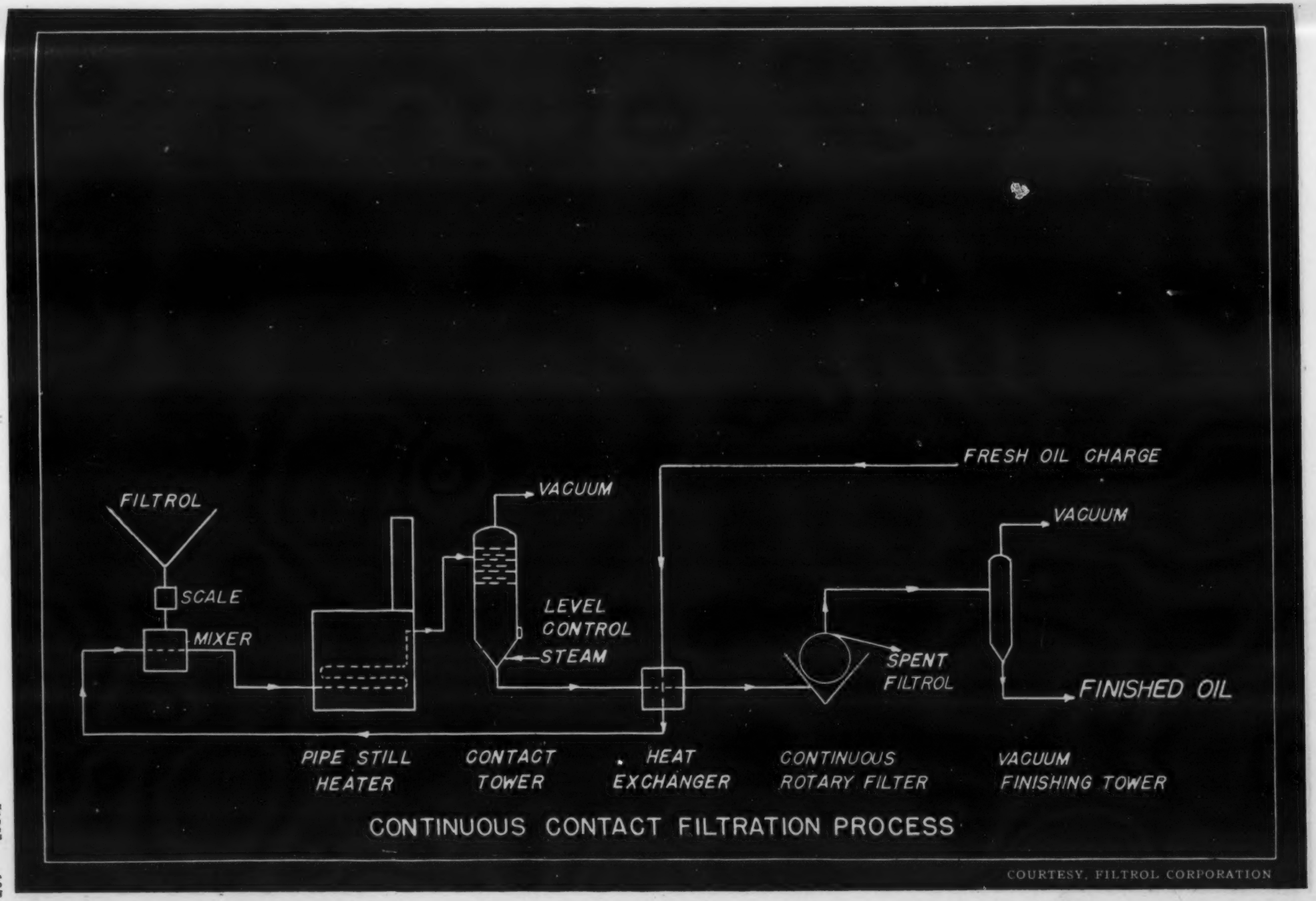
Adaptability to Other Refinery Processes. A contact plant can be used to clarify, decolorize, and/or neutralize lubricating oils whether they have been acid-treated, solvent-extracted or simply distilled. In addition to finishing the normal production of such processes, the contact plant often is used to correct previous faulty refining. Sour oils are neutralized and decolorized in one operation, eliminating caustic neutralization and the accompanying disadvantages of emulsion formation and poor casts.

Low Operating Costs. In a modern contact-filtration unit where adsorbent handling is accomplished by an automatic mechanical system, the operation cost averages approximately $0.08 per barrel for a 2000-barrel-per-day plant.

Maximum Adsorbent Economy. The flexibility of modern design in a contact filtration unit combined with improved adsorbents enables the process to be operated under optimun conditions. This positive control of mixing, heating, filtering and stripping results in maximum adsorbent economy. Generally speaking, activated adsorbent efficiencies increase with temperatures, the optimum point being at or near the flash point of the contact oil.

On the opposite page is a flow diagram of a continuous contact plant presented through the courtesy of Filtrol Corporation, 634 South Spring Street, Los Angeles, California. After Filtrol has been added by means of automatic proportioning equipment to the oil change, the oil-Filtrol mixture enters a pipe-still heater where the maximum contact temperature is obtained in a once-through operation. From the heater, the slurry of oil and Filtrol enters a baffled vacuum stripping tower where a level is maintained regulating the time of contact. Steam sometimes is passed up the tower to facilitate the stripping action. The slurry is drawn continuously from the bottom of the tower, passed through the heat exchanger utilized to heat the fresh-oil charge, and thence to a filter surge tank which acts as a reservoir to feed the continuous precoat rotary filter. The filtered oil from this filter sometimes is charged to a high-vacuum stripper to furnish additional control of flash, odor, oxidation, and emulsion characteristics. The filtered oil finally is pumped through a blotter press which furnishes a polishing step before the oil is transferred to storage. In a few cases where the conditions warrant it, the spent filter cake is extracted with naphtha to recover the small additional quantity of oil soakage.

Filtrol addition, temperatures, rates, etc., are all subject to automatic control in modern installations, hence long uniform runs are currently made without difficulty.



CONTINUOUS CONTACT FILTRATION PROCESS

COURTESY, FILTROL CORPORATION

De Laval Acid Treating Process

THE DE LAVAL SEPARATOR COMPANY

THE De Laval continuous acid treating process as developed by The De Laval Separator Company, 165 Broadway, New York, N. Y., is applicable to the refining of the complete range of oils from cracked naphtha, pressure distillate, kerosene, etc., down to heavy cylinder stocks with viscosities as high as 200 S. S. U. at 210°F.

A feature of this process is the De Laval Acid Sludge Separator of which several types are available. As this centrifugal separator instantaneously and completely separates the acid sludges from the various oils and continuously discharges both oil and sludge, the time of contact between acid and oil may be carefully regulated to the optimum, rather than being determined by the length of time required for the sludge to settle by gravity.

While the problem of acid treating light oils is somewhat different from the refining of heavy lubricating stocks, with the exception of a few details of design, the same equipment is in general, used. The continuous process has been made feasible by the development of a highly specialized centrifugal separator and, of almost equal importance, by the development of properly designed auxiliary equipment to effect the proper treatment of the oil and at the same time to put it in such condition that the sludge can be most efficiently removed by the centrifugal machine. As the acid and oil are carefully metered, continuously mixed and subjected to a predetermined degree of mechanical agitation, the acid is more efficiently utilized with a consequent reduction in acid requirements.

The proper control of contact time between acid and oil in the case of cracked distillates results in lower polymerization losses and lower loss of "anti-knock" rating during acid treatment; in the case of lubricating oil stocks re-solution of the sludge is eliminated with its resultant darkening and instability of the oil. Batch agitators with air agitation are eliminated with their attendant high maintenance, inefficient use of chemicals, inflexible operation and difficult disposal of hard, viscous sludges.

Due to the more complete separation of the acid from the oil, in all cases there is a reduction in refining losses ranging from 25 to 50 percent of those encountered when gravity settling is employed.

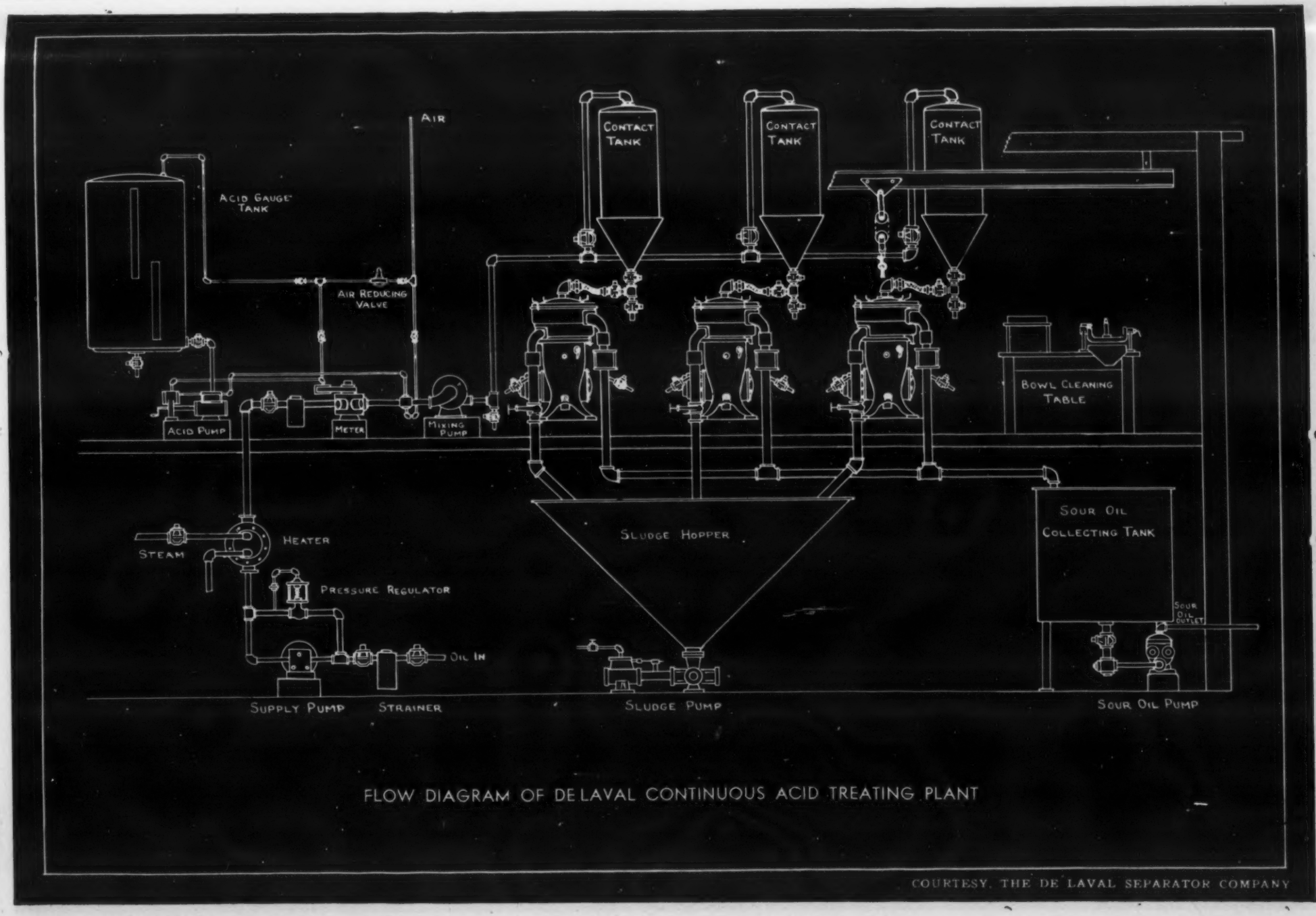
The flow sheet presented here shows the equipment used in the treatment of lubricating oil stocks. With a few exceptions this same flow diagram applies to the acid treatment of cracked distillates.

The operating sequence is as follows: The oil is pumped from the raw oil tank at machine capacity through the tubular steam heater (or tank) and through the proportioning device which maintains the flow of acid into the oil at a predetermined rate. From the proportioning device the mixture passes to the mixing pump where the acid is thoroughly dispersed in the raw oil. This mixing pump discharges into a reaction tank, preferably a closed vessel, where the volume is regulated to achieve the optimum reaction time.

From the reaction tank the mixture of oil and sludge is passed direct to the centrifugal separators. Each centrifuge discharges three components, namely, the sludge-free treated oil, the acid sludge and free acid.

The important points of control affecting the operation are: acid treat, treating temperature, degree of mixing, reaction time, and degree of separation. Of these, the first two are maintained automatically by conventional control devices. The mixing pump is especially designed to give the proper degree of mixing and agitation. The reaction time may vary from a few seconds up to several minutes, seldom exceeding ten, and is dependent upon the nature of the oil being treated and to some extent the degree of agitation in the mixing pump.

Discharge of the sludge and weak acid is accomplished continuously so that there is no necessity of stopping the machines for bowl cleaning. Frequent examinations of bowls in service have failed to reveal any accumulation of the semi-solid, non-plastic material, asphaltic in nature, sometimes encountered in similar operations. Collecting hoppers on the separators are so designed as to facilitate the flow of sludge so that no backing up of the material is experienced. The machines are shut down for inspection at intervals of a week or so. The sludge discharged from the centrifugal separator is less viscous and can therefore be disposed of more readily than that drawn from batch agitators.



FLOW DIAGRAM OF DE LAVAL CONTINUOUS ACID TREATING PLANT

COURTESY, THE DE LAVAL SEPARATOR COMPANY

Delayed Coking Process

THE M. W. KELLOGG COMPANY

DELAYED Coking is a continuous thermal-cracking process designed to produce clean (low Conradson carbon) catalytic-cracking feed stock from any type of reduced crude. A by-product of the process is a high grade of petroleum coke useful as a raw material in chemical industries.

The overall effect of the process on modern refinery operation is to increase the yields of motor gasoline and diesel fuel per barrel of crude oil processed, and to reduce the percentage of fuel oil.

There are at present about 13 Delayed Coking units operating or building in this country with a combined capacity of approximately 40 million barrels of reduced crude yearly.

Feed to the unit is usually a heavy reduced crude. After heat exchange with the hot products the feed is pumped to the base of a bubble-tower fractionator where any light components are flashed off. The bottoms of this bubble tower are pumped to a pipe furnace in which they are quickly heated to about 920° F. and discharged at a pressure of 10-70 psig into insulated coke drums to coke by their contained heat. The actual number of drums required is determined by the feed rate and carbon-forming characteristics of the reduced crude. At any given time half of the drums are coking and the other half are decoking. A specially designed transfer manifold utilizing an unusual switch valve makes it possible to feed any drum without coking liquid in lines or interrupting furnace operations.

A mixture of liquid and vapor enters the bottom of the coke drums but only vapor leaves the top, all liquid being converted in the drums to either coke or vapor. The vapors leaving the drums are fractionated in a bubble tower, producing gas and gasoline overhead and withdrawing light, medium and heavy gasoil as side streams. The heaviest gasoil, blended with the flashed residuum, is returned to the furnace.

The above description is applicable to recycle operation. However, Delayed Coking units are also operating on a single-pass basis.

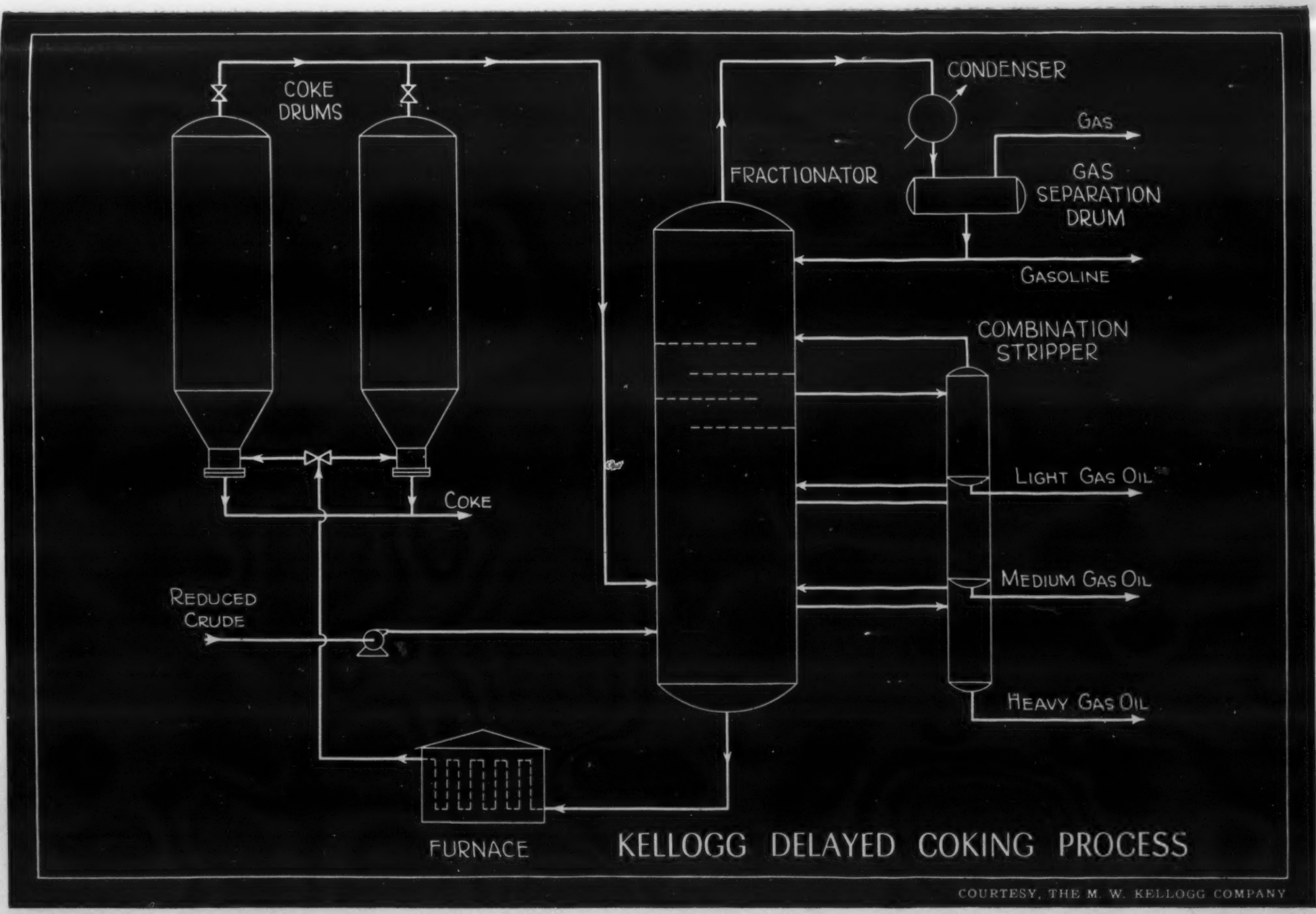
Removal of coke from the drums is accomplished hydraulically by means of high impact-producing jets incorporated in special boring and cutting tools. The hydraulic method has been perfected to a point where the drums are cleaned speedily and safely and the coke transported to storage in a continuous mechanical operation.

Delayed Coking yields on a typical charging stock are given in the following table:

Table of Delayed Coking Yields and Product Inspection

Reduced Crude Charge	
Weight %	100
Gravity, °API	21.0
Conradson Carbon, %	6.0
Products	
1. **Stabilized Gasoline**	
Volume %	16.8
Gravity, °API	61.2
ASTM Distillation, °F.	
IBP	125
10%	175
50%	285
90%	365
EP	400
Reid Vapor Pressure, psi	4.0
CFRM Octane No.—Clear	60.0
2. **Gas Oil**	
Volume %	71.5
Gravity, °API	29.0
Conradson Carbon, %	0.03
3. **Dry Gas**	
Weight %	4.9
4. **Petroleum Coke**	
Weight %	12.9
Ash, %	0.15

The M. W. Kellogg Company, New York, has been appointed licensing agent for this process for the United States and foreign countries.


COKE DRUMS
CONDENSER
GAS
FRACTIONATOR
GAS SEPARATION DRUM
GASOLINE
COMBINATION STRIPPER
LIGHT GAS OIL
COKE
MEDIUM GAS OIL
REDUCED CRUDE
HEAVY GAS OIL
FURNACE
KELLOGG DELAYED COKING PROCESS
COURTESY, THE M. W. KELLOGG COMPANY

Diethylene Glycol Process

FOR DEHYDRATING NATURAL GAS

NATURAL gas normally contains a certain amount of water vapor. Unless the water content is reduced so that no condensation occurs under the conditions of temperature and pressure existing in natural gas transmission lines, operating difficulties are likely to develop. Moisture causes internal corrosion and rust accumulations. Water condensing at the low points in the transmission line must be drained off from time to time to prevent stoppage and freezing. In addition, at high pressures and in the presence of liquid water, solids known as natural gas hydrates may form, which resemble snow and build up in the line, seriously interfering with its capacity. At high pressures these hydrates may have melting points as high as 50-60° F. It is therefore apparent that atmospheric temperature conditions do not have to be particularly severe before difficulties may be encountered.

For the above reasons it has been necessary to provide for dehydrating natural gas to reduce the dew point before sending the gas through long high pressure transmission lines which are subjected to low atmospheric temperatures. Dehydration to a dew point below the minimum temperature to which the line will be subjected in the territory through which it passes has the following advantages:

(1). Maintenance at all times of full line capacity which would otherwise be reduced due to the formation of hydrates and water condensate. (2). Elimination of the necessity of blowing the drips to remove condensate. (3). Elimination of regulator freeze-ups. (4). Reduction of internal corrosion of the pipe line.

Natural gas dehydration is generally accomplished by contacting the gas with an hygroscopic liquid. Refrigerated calcium chloride water solutions have been used for this purpose. The cycle of operation when using diethylene glycol is basically the same as for brine. Dew point depressions up to 60° F. can be secured with concentrated diethylene glycol without refrigeration, which is necessary when correspondingly low depressions are to be secured with brine solutions. Refrigeration equipment materially increases plant investment. Diethylene glycol is not corrosive, and has a low freezing point so there is no possibility of solidification of the solution in the system.

Properties of Diethylene Glycol

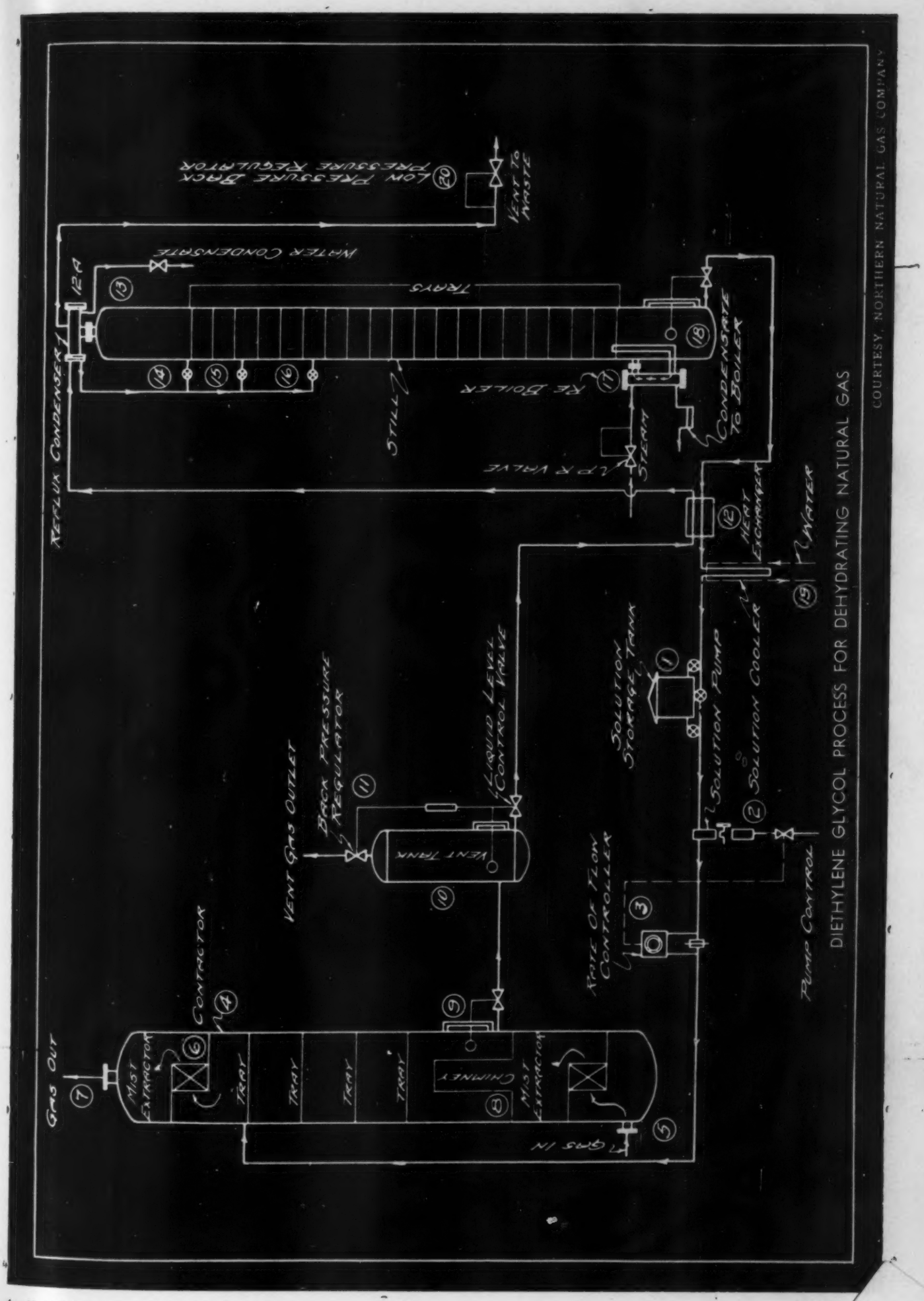
Formula	$HOCH_2CH_2OCH_2CH_2OH$
Molecular Weight	106.08
Boiling Point	244.5°C. (472.1°F.)
Freezing Point	—6.5°C.
Specific Gravity	1.1185 20/20°C.
Weight Per Gallon	9.308 lb. 20°C.
Viscosity	0.38 poises 20°C.
Vapor Pressure	0.015 mm. Hg 20°C.
Specific Heat	0.5509 20°C.
Latent Heat of Evaporation	150 cal./g. at B. P.
Coefficient of Expansion (per °C.)	0.000635 at 20°C.
	0.000650 at 55°C.

Operation of the plant, as shown in the flow diagram on the opposite page, is as follows: Cooled diethylene glycol containing a small amount (generally less than 10 percent) of water depending on the final dew point desired is picked up from the solution storage tank (1) by a pump (2) capable of pumping against the pressure at which the gas is to be treated. The solution flows through a flow controller (3) which controls the speed of the pump and into the top of the tray type contactor (4). Gas enters the bottom of the contactor through a mist extractor to remove mineral seal oil, etc. The contactor is equipped with bubble cap trays, the solution descending counter-current to the ascending natural gas. The gas leaves the contactor through mist extractor (6) and outlet connection (7) into the transmission line. The solution which has absorbed water from the gas is collected on the chimney plate (8). The liquid level on the chimney plate is controlled by the flow controller (9) and flows into a vent tank (10) provided with a back pressure regulator (11) which maintains sufficient pressure in the vent tank to force the liquid through the heat exchangers (12) and the reflux condenser (12-A) into the top of the still (13). Natural gas released in (10) is vented through the vent gas outlet.

Glycol solution passes through the reflux condenser (12-A) into the still through valves 14, 15 or 16 as desired, generally through one of the lower openings. The solution flows down over bubble cap trays in the still column and into the lower portion of the re-boiler (17) where it is heated by closed steam coils and is returned to the kettle (18) in the lower part of the still. Water remaining in the heated solution is vaporized in the kettle and passes through the bubble cap trays of the still column. This serves to heat the descending solution and separates the diethylene glycol from the water vapor, thus eliminating loss of diethylene glycol in the still. Any natural gas or uncondensed water vapor in the reflux condenser (12-A) are vented through the back pressure valve (20). Excess condensate in the reflux condenser is drained through the condensate line to the sewer. The solution in the kettle (18) having been substantially denuded of water passes through the heat exchangers (12) in counter-current relation to the weak solution from the vent tank, and thence through a cooler (19) to storage tank (1). Replacement solution is added to the system when necessary, at this point. The cycle of operation described is continuous.

In commercial plants the total over-all loss of diethylene glycol averages .05 gallons per million cubic feet measured at atmospheric pressure.

Diethylene glycol is manufactured by the Carbide and Carbon Chemicals Corporation, 30 East 42nd Street, New York, N. Y.


DIETHYLENE GLYCOL PROCESS FOR DEHYDRATING NATURAL GAS
COURTESY, NORTHERN NATURAL GAS COMPANY
GAS OUT
GAS IN
MIST EXTRACTOR
TRAY
TRAY
TRAY
TRAY
CHIMNEY
MIST EXTRACTOR
CONTACTOR
VENT GAS OUTLET
BACK PRESSURE REGULATOR
VENT TANK
LIQUID LEVEL CONTROL VALVE
RATE OF FLOW CONTROLLER
SOLUTION STORAGE TANK
SOLUTION PUMP
SOLUTION COOLER
PUMP CONTROL
HEAT EXCHANGER
WATER
CONDENSATE TO BOILER
STEAM
LP R VALVE
STILL
RE BOILER
TRAYS
REFLUX CONDENSER
WATER CONDENSATE
LOW PRESSURE BACK PRESSURE REGULATOR
VENT TO WASTE

Donnelly Cracking Process

DONNELLY PROCESS CORPORATION

DONNELLY PROCESS CORPORATION, Chicago, Illinois, licenses "Coil and Quench Cracking Process" which has an advantage in the production of anti-knock (high octane rating) gasoline, so much in current popular demand. The inhibition of carbon formation by control of temperature and time factors during cracking and separation is covered by patents and applications owned by this company and is fundamental to long operating cycles, and production of low B.S. merchantable residuum. The wide use of these principles in modern cracking technique attests to their effectiveness. The processing principles employed in this process provide a basis for cracking unit design having the widest possible range of operating flexibility as to temperatures, pressures, charge stocks treated, and products made.

Donnelly units are designed to meet each individual refiner's requirements as to capacity and type of unit. The unit may be constructed as small as a simple 250-barrel daily charge capacity unit having a single heating coil for cracking wide boiling range cycle oil, producing gas, gasoline and fuel oil. The unit may be constructed as large as a 20,000-barrel daily charge capacity combination crude topping and cracking unit having multiple heating elements for simultaneous differential time, temperature and pressure cracking of several selected oil fractions, producing gas, high octane rating gasoline, naphthas, tractor distillates, furnace distillates, road oils, fuel oils, and other residuals.

The accompanying flow diagram illustrates in a general way one method of application of these processing principles to selective differential cracking. In this specific case the fresh charge oil, consisting of topped crude oil, wax and other heavy distillate, is preheated to incipient or low rate cracking by indirect heat interchange with vapors passing from the vapor separator to the bubble tower and by hot gases in the convection section of the furnace and is then commingled with the effluent from the high boiling cycle oil cracking coil. The amount of cracking of the charge oil required will govern the amount of cracking to be done in the high boiling cycle oil coil. If distillation with little or no cracking of the charge oil is desired, the operation will be conducted so that cracking in the coil of the high boiling cycle oil fraction will be extensive, that is, approximating maximum gasoline formation, without free carbon formation, and the mixture of cracked oil and fresh charge oil will be passed immediately to the vapor separator. If cold test reduction or viscosity cracking of the charge oil is required, the operation will be conducted so that cracking in the coil of the high boiling cycle oil fraction, prior to commingleme with the fresh charge oil, will be less extensive than in the previous example. In this case the cracking treatment will be continued after commingleme by passing the mixture through a series of well insulated coils with detachable end fittings, or through a drum designed to provide the proper time element to carry the cracking to the desired amount. After this treatment, the mixture is introduced into the vapor separator.

The proper operation of the vapor separator is a fundamental factor in production of merchantable fuel oil and the inhibition of carbon formation in the chamber. Accordingly, the Donnelly process provides for sufficiently cooling the cracked products either before, simultaneous with, or subsequent to separation, to prevent residual fractions from being reduced to free carbon or coke during separation. Several means for accomplishing this cooling are shown on the diagram.

The vapors separated in the vapor separator free from cracked residual fractions pass through a heat exchanger, then to a bubble tower where they are fractionated and separated into two liquid fractions each having different boiling ranges, the higher boiling fraction accumulating in the bottom of said tower, and the lower boiling fraction being withdrawn to a separate accumulator. Each such fraction is delivered by a hot oil pump to its respective cracking coil, where it is treated to conditions of time, temperature and pressure best suited to its characteristics to obtain the desired products. For heat economy, as shown on the diagram, the low boiling cycle oil fraction before entering its cracking coil passes in heat exchange with its own outlet.

The vapor uncondensed in the bubble tower is passed through a condenser, into a separator, from which high octane rating gasoline and fixed gases are withdrawn.

The liquid separated in the vapor separator is withdrawn under reduced pressure into a flash drum, from which the resulting vapor is withdrawn, condensed, accumulated and returned to the system, and the unvaporized portion, constituting residuum (fuel or road oil) is withdrawn, cooled and delivered to storage. A portion of this residuum may be returned to the vapor separator or the flash chamber to control the temperature thereof.

The flash drum shown on the diagram is an extension of the vapor separator skirt, and may be constructed of lighter material than the vapor separator.

With proper instrumentation the unit's operation may be practically automatically controlled.

DONNELLY PROCESSING
FLOW DIAGRAM
COURTESY, DONNELLY PROCESS CORPORATION
GAS
GASOLINE
STEAM
LOW BOILING FRACTIONS
BUBBLE TOWER
HOT OIL PUMP
STEAM
HIGH BOILING FRACTIONS
TUBE TYPE HEAT EXCHANGERS
FRESH CHARGE
VAPOR SEPARATOR
FLASH DRUM
FLASH DISTILLATE ACCUMULATOR
RESIDUUM TO STORAGE
COIL TYPE HEAT EXCHANGER
VISCOSITY CRACKER
TUBE STILL

Dual-Flo Distillation Process

BORN ENGINEERING COMPANY

THE refiner is often faced with the problem of processing crude oils differing widely in properties and producing products differing considerably in specifications. Some refineries have attempted to solve this problem by "batching" the crude, that is, running one type of crude for a period and then following it with another type. However, this necessitates considerable adjustment of furnace temperature and tower conditions, and results in costly delays.

The Born Dual-Flo crude distillation unit processes two crude oils separately and simultaneously. By this method, the refiner can process two crude oils of entirely different specifications simultaneously and yet take advantage of getting the maximum yield of specification product, plus operating economy and flexibility.

As an example, a refiner has two types of crude oil, one producing straight-run gasoline with a high octane rating suitable for aviation base stock and another crude with a low octane straight-run gasoline. If these crudes were blended and distilled in a single operation, it would not permit the refiner to realize the advantage of the higher octane product. Another example of the utility of the Dual-Flo unit is when the refiner has both paraffin base and asphalt base crude oils and is desirous of producing both lubricating oils and asphaltic products. By using this unit the various products could be separately obtained at the same time.

The attached flow diagram illustrates the general arrangement of the unit. The following results taken from a commercial operation of one of these units illustrates the processing:

"Charge B, a mixed crude having an average gravity of 35° API, 0.7 percent sulfur and 125-130 pounds of salt per 1000 barrels of crude is processed at the rate of 2500 barrels per day. Salt is taken out at around 200° F. and 250 psi in the settler. The overhead from this crude, a gasoline having an octane value of approximately 55, is used for blending to motor fuel. Other yields include the side cuts of tractor distillate, light and heavy gas oils and bottoms.

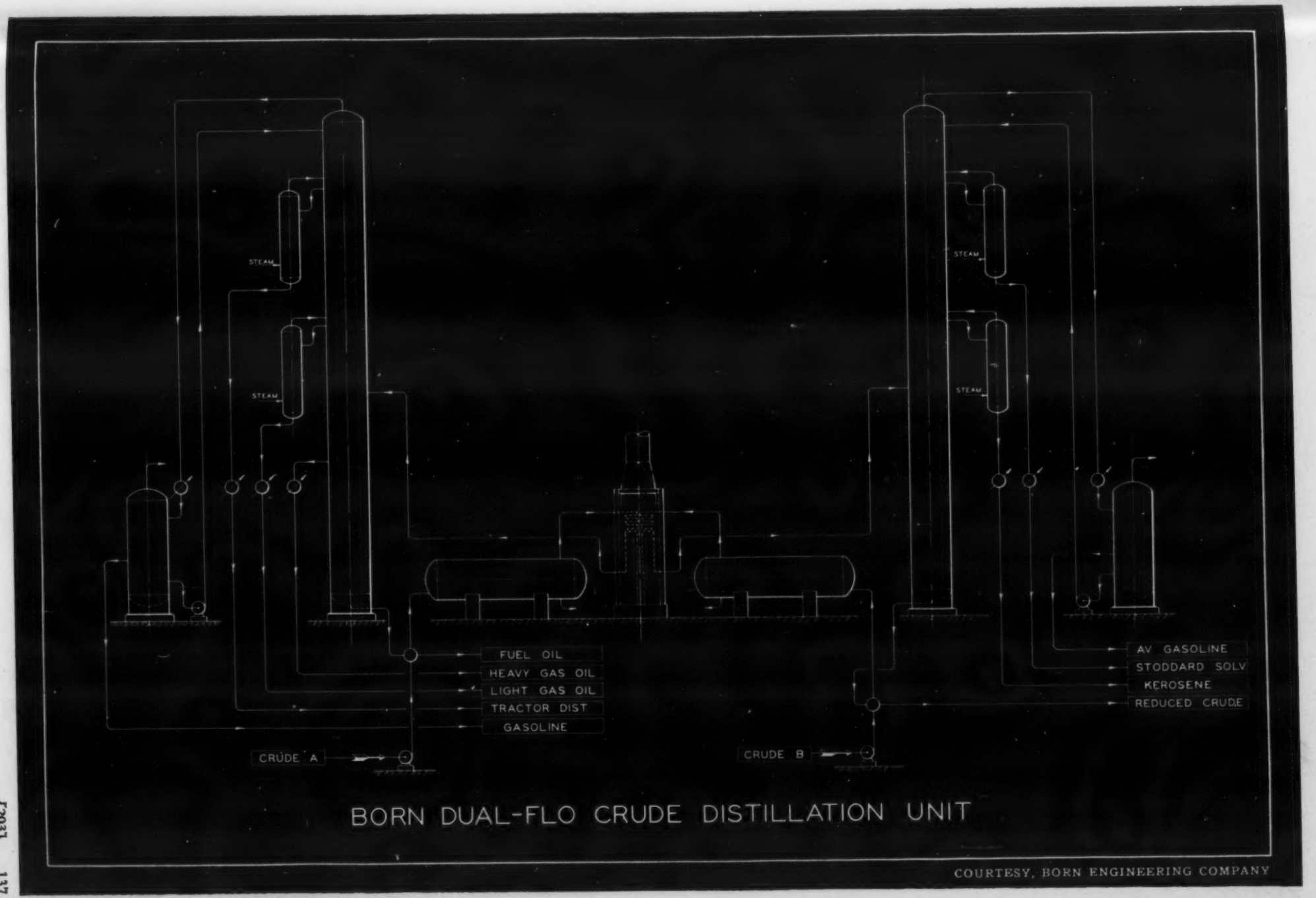
"A Cromwell-sand crude is used for Charge A which averages about 30.5° API gravity and contains approximately 200 pounds of salt per 1000 barrels and 0.02 percent sulfur. The charging rate for the Cromwell-sand crude is 2500 barrels per day. Gasoline from this charge stock has an octane rating of from 72 to 73 and when blended with 3 c.c. of lead produces a 90-91-octane fuel. Stoddard naphtha, kerosine and the residual product comprise the other yields from this section of the unit.

"Residual yields from both sections are mixed for a cracking stock. By combining the tractor distillate and the intermediate cuts a 350°-540° F. boiling range butadiene adsorption stock is obtained. Flexibility of the unit is illustrated in an operation when one section was rerunning pressure-distillate for an 80-octane all-purpose gasoline at the same time crude was being processed to yield an overhead naphtha."

Centralization of control and other facilities common to both sections of the unit contribute to operational convenience and present a balanced plant layout. The Born Dual-Flo Heater has two combustion chambers and a single gas-duct system. The furnaces have floor-fired burners, giving close control and uniform heat distribution. Both heater coils pass from a common convection zone into their respective locations in the separate combustion chambers. Heating-coil temperatures are controlled automatically. Because each section of the heater coil is equipped with a temperature controller, the amount of heat to separate coil systems may be varied independently when desired.

Maximum flexibility of operation is accomplished by arranging the process piping so that the fractionators and the other equipment in either section of the process may be cut in or cut out as desired when running the unit on the different charges which are to be processed.

The flow diagram on the opposite page as well as this description of the process were provided by Born Engineering Company, Tulsa, Oklahoma, designer of the unit.



BORN DUAL-FLO CRUDE DISTILLATION UNIT

COURTESY, BORN ENGINEERING COMPANY

Duo-Sol Solvent Extraction Process

MAX B. MILLER & COMPANY, INC.

THE Duo-Sol Process developed by the Max B. Miller & Company, Inc., 501 Fifth Avenue, New York, N. Y., and presented here through the courtesy of that company, is a solvent refining process producing from any stock a maximum yield of lubricating oil of any quality desired.

The basic principle of the Duo-Sol Process is the simultaneous use of two practically immiscible solvents in an extractor system having one or more extract stripping stages in addition to the conventional raffinate stripping stages. Propane is employed as a paraffinic solvent and Selecto, a blend of phenol and cresol, is the selective naphthenic solvent. Separation of the two layers in each extraction stage is accomplished by gravity.

The Selecto layer takes into solution the asphaltic unstable and otherwise undesirable fractions of the charge stock. The propane layer carries the stable paraffinic lubricating fractions. The two solvents flow countercurrently through the extractor system, the stock charge being introduced at an intermediate stage. As is shown in the flow sheet, the extract layer is contacted with an increasingly concentrated propane solution in flowing from stage 3 to stage 1, and is thereby stripped of any paraffinic fractions it might contain. In a similar manner, as the propane-raffinate layer flows in the opposite direction from stage 3 to stage 7, it comes into contact with a more and more concentrated solution of Selecto, thus being stripped of its asphaltic oil content.

Advantages of the use of a two-solvent system employing an extractor containing an extract stripping section as well as a raffinate stripping section, are as follows:

(a) Greater Flexibility. The flexibility of the Duo-Sol process permits the treatment of distillates or residuums from any type of crude, producing a paraffinic oil which, when dewaxed to a zero pour, will in most cases have a viscosity index of 100 or higher. On the other hand, the Duo-Sol process permits the production of the maximum yield of lower quality finished stocks if such an operation is desirable.

(b) Low Overall Operating Costs. The overall cost of producing the finished lubricating oil is greatly reduced since acid treatment is entirely eliminated and clay treatment is cut to a minimum. Because of the ability of the process to handle any type of charging stock, the costs of primary distillation and final fractionation into blending stocks for a full line of motor oils are curtailed.

(c) High Quality of Raffinate. The raffinates obtained by this process are not only high in viscosity index but are exceptionally low in carbon residue, resistant to oxidation, stable as to color, and when lightly filtered will meet any emulsification tests.

(d) Maximum Selectivity. The selectivity of this process is excellent as is shown by the specific gravities of the extracts which range from 1.01 for a light distillate to 1.098 or higher for a heavy residual charging stock.

The number and capacity of the existing Duo-Sol installations indicate that the above mentioned advantages are obtained in actual commercial practice. At present there are in operation in this country six plants having a combined charge capacity of 23,500 barrels per day. There are also nine plants abroad having a total charge capacity of 13,000 barrels per day.

The flexibility of the process is demonstrated by the fact that in the space of a month and a half one of the commercial plants has treated satisfactorily a light and a heavy Colombian distillate having gravities of 21.2° A.P.I. and 19° A.P.I. respectively, an Oklahoma City distillate having a gravity of 28.8° A.P.I., and an Oklahoma City residuum having an A.P.I. gravity of 21.0°.

Solvent recovery from each layer is accomplished by vaporization of propane in a high pressure tower and the stripping of Selecto from each layer in a low pressure tower. Suitable exchange is provided for the recovery of the maximum amount of heat. Separation of the stripping steam from the Selecto is accomplished by taking a constant boiling mixture overhead from Tower W-1 and decanting in Tanks W-2 and W-3.

Some typical results obtained by this process are tabulated below. As can be seen, charging stocks ranging from a light transformer oil to the heaviest crude bottoms can be readily handled in the same plant.

Characteristics of Charging Stock	Transformer Oil	Mid-Continent Distillate	Oklahoma City Residuum	South Texas Tar
Gravity A.P.I.	24.5	25.4	24.2	1.001 Sp. Gr.
Vis. @ 210° F. S.S.U.	64.5 @ 100	48	78	2700
Flash ° F.	300	430	400	.....
Raffinate (Dewaxed):				
Gravity A.P.I.	27.0	31.1	29.2	26.2
Vis. @ 210° F. S.S.U.	63 @ 100	97 @ 130	60	182.0
Pour	0° F.	0° F.	0° F.	0° F.
V. I.	.....	98	100	95.5
Color NPA (before filtering)	1	3	8—	7—
Yield, percent (Duo-Sol)	88.0	78.3	77.8	27.0
Specific Gravity of Extract	1.012	1.015	1.032	1.04

DUO SOL SOLVENT EXTRACTION PROCESS
COURTESY, MAX B. MILLER & COMPANY, INC.
LEGEND
NORMAL PROCESS LIQUID LINES
NORMAL PROCESS VAPOR LINES
PROCESS WATER LINES
STRIPPING STEAM
EXTRACT SELECTO EVAPORATOR
EXTRACT PROPANE EVAPORATOR
NAPHTHENIC LAYER
FURNACE
STEAM CONVERTER
EXTRACT COOLER
FINISHED EXTRACT
EXTRACT RUN TANK
SELECTO VAPORS
SELECTO REFLUX
PROPANE VAPORS
PRE-HEATER
WATER CONDENSER
SELECTO COOLER
EXTRACT LAYER
PROPANE REFLUX
PROPANE HEATER OR COOLER
SLOP TANK
CRUDE MIX COOLER
MIXER
CRUDE CHARGE
START HERE
CRUDE TANK
PROPANE CHARGE
RAFFINATE PROPANE EVAPORATOR
PROPANE VAPOR
RAFFINATE LAYER
SELECTO LIQUID
SELECTO WATER
EXTRACTORS
PROPANE CONDENSER
TEST CONDENSER
TO REFINED GASOLINE
PROPANE STORAGE
PROPANE COMPRESSION
SELECTO CHARGE
SELECTO TANK
SELECTO COOLER WATER
SELECTO FREE WATER
RAFFINATE SELECTO EVAPORATOR
STEAM GENERATOR
RAFFINATE COOLER
RAFFINATE RUN TANK

Dow Styrene Process

THE DOW CHEMICAL COMPANY

THE Dow Styrene Process was developed during the early 1930's at The Dow Chemical Company's Research Laboratories in Midland, Michigan, as a step in the manufacture of the plastic polystyrene. The wartime synthetic rubber program resulted in construction of four plants which furnished styrene for GR-S rubber. Although the process was licensed during the emergency, it is not generally available.

The process in brief is the catalytic dehydrogenation of ethylbenzene to styrene at a conversion per pass of about 37 percent and an ultimate yield of 90 percent, with subsequent vacuum distillation of the crude styrene.

Since reduced pressure favors the dehydrogenation reaction, steam is fed at a rate of 2.6 pounds per pound of ethylbenzene. About 90 percent of this steam quantity is heat-exchanged to a temperature of 385° C. before entering the superheating furnace, which produces steam at 710° C. The remaining 10 percent of the steam is mixed with ethylbenzene before being passed through a vaporizer, attaining a temperature of 160° C. before heat exchange with hot reactor product. At 520° C. the ethylbenzene vapors meet superheated steam at 710° C. The resulting temperature at the bottom of the catalyst bed is 630° C. This temperature point is used for controlling the reaction system. Reactor product at 565° C. is cooled by heat exchange, first with incoming ethylbenzene, and then with steam. From this point a spray-type desuperheater lowers the crude product temperature to 105° C. and condenses out tars formed at high temperatures. Vapors then enter the final condenser, where steam, styrene, ethylbenzene, benzene, toluene, and small amounts of tar are liquefied. Vent gasses containing hydrogen, carbon monoxide, carbon dioxide, methane, ethane, plus some of the aromatics pass overhead to a refrigerated recovery system.

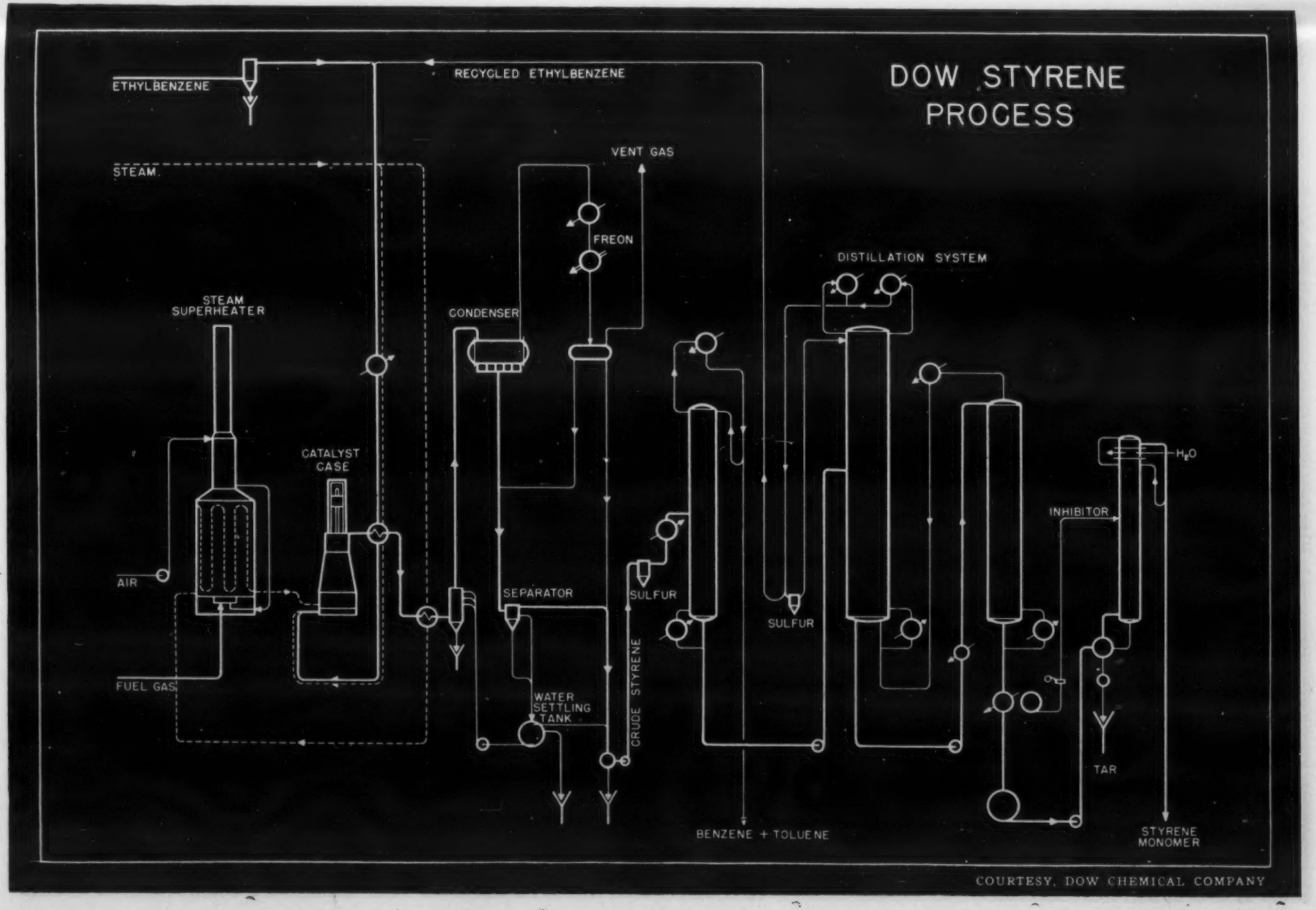
The materials condensed go to a gravity separator, where the hydrocarbons are decanted from the water phase. After further settling, part of the water is recycled to the sprays of the desuperheater, and the rest is discharged to a disposal system. The hydrocarbons pass to another settler, where insoluble tar can drop out along with entrained water. Crude styrene then is pumped to storage. An average composition for this stream by weight is:

	Percent
Styrene	37.0
Ethylbenzene	61.1
Toluene	1.1
Benzene	0.6
Tar	0.2

The final step is the purification of the crude dehydrogenated material containing about 37 percent styrene. The fractionation requirements are rather strict since the normal boiling points of ethylbenzene and styrene differ by only 9° C. Other factors complicate the distillation step to an even greater extent. Distillation of styrene at its atmospheric boiling point (145.2° C.) is impossible because of rapid formation of polystyrene. At 30 mm. Hg. absolute, the boiling point of styrene is 54° C., and the polymerization rate is less than 0.1 percent per hour. Running under a vacuum, then, is necessary. Even though a low distillation temperature is achieved, the normal irregularities of operation make further precautions necessary. Very small quantities of certain foreign materials catalyze the polymerization reaction greatly. To guard against such possibilities, elemental sulfur is dissolved in styrene to act as a polymerization inhibitor. Only by a combination of vacuum operation, suitable inhibition of styrene, and special column design can styrene be distilled successfully. Relatively trouble-free operation can be had by distilling inhibited styrene in a system which never allows the concentrated monomer to exceed 90° C.

In following the flow further, crude styrene passes through a pot containing sulfur. Enough sulfur is dissolved to act as a polymerization inhibitor. After preheating, the feed enters the 30-plate benzene-toluene column where benzene and toluene pass overhead at 57° C. The top of this column operates at 175 mm. Hg., and a reflux ratio of 12 to 1 is employed. Since styrene is still diluted with ethylbenzene and tar, a temperature of 96° C. is allowable at the bottom. Sulfur passes down the column and effectively inhibits the stripping zone. Benzene and toluene from all four finishing blocks then pass to a benzene column of standard construction operating at atmospheric pressure. It contains 40 plates and operates with a reflux ratio of 3 to 1. Benzene, thus purified to less than 0.1 percent toluene content, is recycled to the ethylbenzene plant for alkylation. The bottoms from this column contain toluene and traces of ethylbenzene. Toluene with a 1° C. boiling range is obtained as product from the 35-plate toluene column. Ethylbenzene, styrene, and tar then pass to the primary ethylbenzene column. At this stage comes the rather difficult separation of ethylbenzene from styrene. A single column of 70 plates would give a bottom pressure of 225 mm. Hg. and a condition where highly concentrated styrene would be boiled at 104° C. This temperature would not be advisable even in the presence of sulfur. To overcome this difficulty the separation is split into two steps: the primary ethylbenzene column, which acts as the top 38 plates, and the secondary ethylbenzene column acting as the bottom 32 plates. Vacuum jets and cooling water keep the top of each at 35 mm. Hg., and thus, the bottom temperatures can be held at a safe level of 90° C. The reboilers of these stills are short-tube vertical units designed for a very low liquid head in the tubes. Steam at 5 pounds gauge is on the shell side. Low t's in the reboilers are necessary to prevent high skin temperatures. The columns are designed for a low pressure drop and many precautions are taken to prevent the inward leakage of air. The upper part of the primary column is protected against polymer formation by the addition of sulfur to ethylbenzene reflux. This primary column takes recycle ethylbenzene containing about 1 percent styrene overhead and to the feed of the dehydrogenation step. Bottoms from the primary are pumped through a cooler to the top of the secondary column. The overhead from the secondary column is condensed, then flows by gravity to the reboiler of the primary. Condensation of this stream has proved desirable from the control standpoint. Bottoms from the secondary column are cooled to prevent polymerization and then pass to a batch-still charge tank which is held at 35 mm. Hg. pressure. Packed batch styrene finishing stills are used to remove tar and sulfur from the styrene. Batch operation permits somewhat easier handling of the residue and also gives greater flexibility to the system. A styrene finishing still runs at varying reflux ratio depending on the purity of the overhead product. Product purity is recorded continuously on a strip chart by a specially designed infrared analyzer. Top temperature is 57° C. at a pressure of 35 mm. Hg. Since the upper part of the column operates with little sulfur present, a solution of para-tertiary-butyl catechol in styrene is pumped into the reflux to this still by means of a proportioning pump. Five parts per million of inhibitor are effective in preventing excessive polymer formation. The residue from these columns is drained while hot into dump buckets. This heavy material, consisting of tars formed in the dehydrogenation step, styrene-sulfur compounds, elemental sulfur, polystyrene, and styrene then are burned. Before shipment, para-tertiary-butyl catechol is added to bring the concentration to a minimum of 10 ppm. Shipment of the finished material is made in insulated tank cars. Styrene is pumped through a refrigerated cooler to these cars to provide a loading temperature below 20° C. With these precautions, the probability of polymer formation in normal tank-car shipment is exceedingly small.

The process described is highly instrumented. The on-stream time of the units is quite high—98 percent or more.


DOW STYRENE PROCESS
ETHYLBENZENE
RECYCLED ETHYLBENZENE
STEAM
VENT GAS
FREON
DISTILLATION SYSTEM
STEAM SUPERHEATER
CONDENSER
CATALYST CASE
H_2O
INHIBITOR
AIR
SEPARATOR
SULFUR
SULFUR
FUEL GAS
WATER SETTLING TANK
CRUDE STYRENE
TAR
BENZENE + TOLUENE
STYRENE MONOMER
COURTESY, DOW CHEMICAL COMPANY

Edeleanu Process

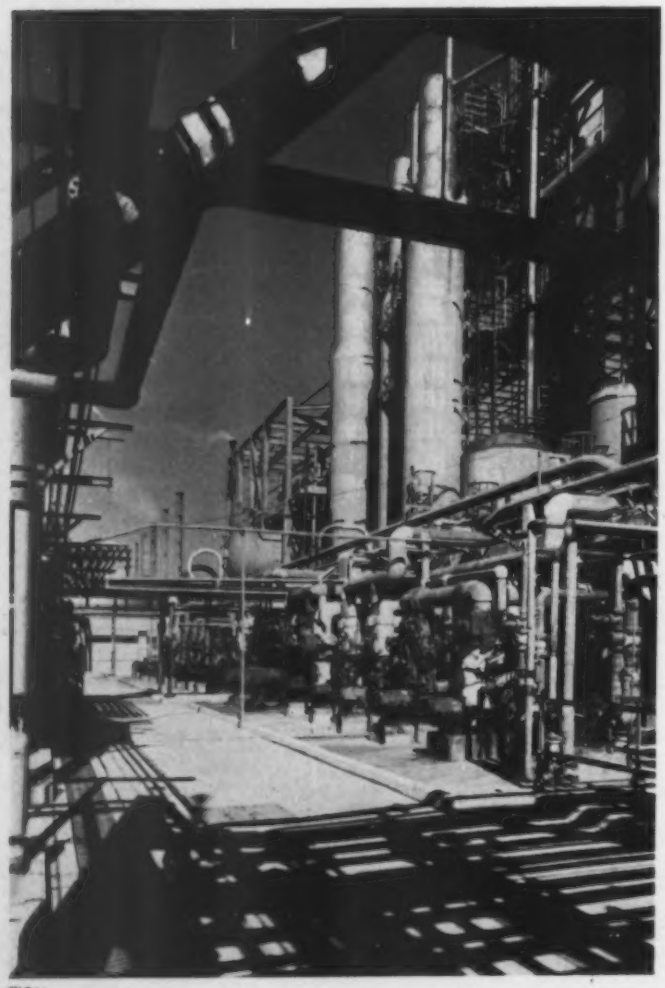
E. B. BADGER & SONS COMPANY

THE Edeleanu Process, employing liquid sulfur dioxide as a solvent, has been used commercially since 1911 for refining petroleum products. Its first application was for the production of high-grade kerosines from highly aromatic and sulfur-bearing crude kerosine distillates. This advance over heavy acid treatments with their attendant high loss of hydrocarbons led to its eventual application to the entire range of petroleum products. The Edeleanu Process is unique insofar as it is the only solvent refining process by which all fractions of mineral oils may be extracted. The low boiling point of sulfur dioxide (14° F.) makes the problem of recovering the solvent relatively simple even though hydrocarbons in the gasoline boiling range are treated.

The flow diagram on the opposite page, presented through the cooperation of E. B. Badger & Sons Company, 75 Pitts Street, Boston, Massachusetts, shows the essential equipment in a typical Edeleanu Unit. A study of the flow sheet shows that the plant consists of the following main sections:

(1) Counter-current extraction tower for contacting oil and solvent.

(2) Equipment for the recovery of the small amount of solvent which dissolves in the refined oil.



(3) Equipment for recovery of the bulk of the solvent which leaves the extraction equipment in the extract.

(4) Equipment for cooling oil and solvent, compressing, recondensing and dehydrating the solvent.

Conventional equipment is employed and a simple system for continuously dehydrating the liquid sulfur dioxide eliminates corrosion. With minor exceptions, ordinary steel equipment is used throughout the plants.

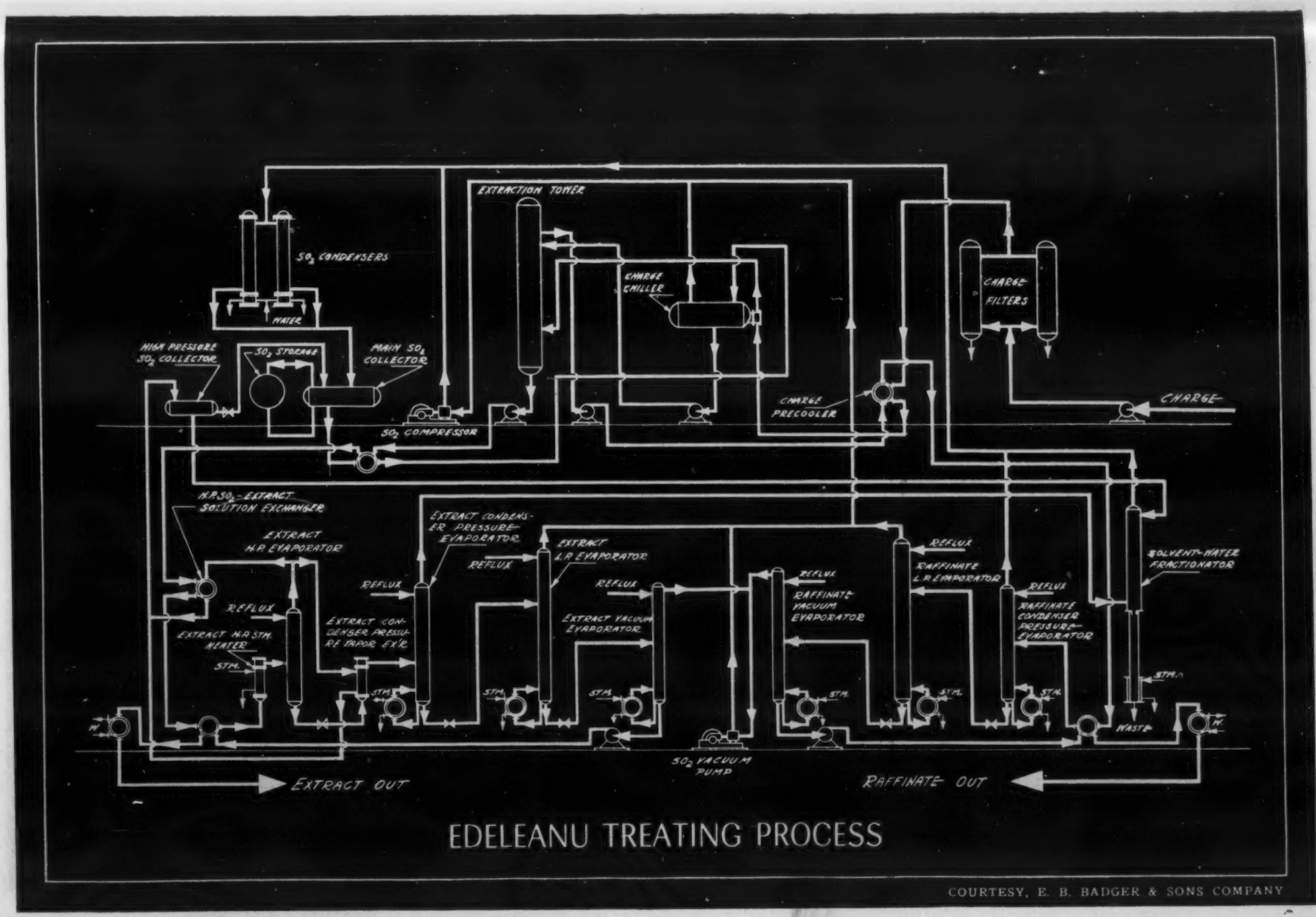
Liquid sulfur dioxide preferentially dissolves aromatic hydrocarbons. It has a less definite preference for naphthenic hydrocarbons and little solubility for paraffines. The degree to which naphthenes and paraffines are dissolved depends upon the temperature of extraction and the boiling range of the stock being treated. The lower the extraction temperature, the less paraffines and naphthenes will be dissolved. The lower the boiling range of the material being extracted, the greater will be the solubility of the SO_2 for paraffines and naphthenes. Generally, therefore, lower-boiling materials are treated at lower temperatures than higher-boiling ones.

The extraction by liquid sulfur dioxide of naphtha is commercially accomplished at temperatures varying from +10° F. to —60° F. The raffinates will be practically free of aromatics and frequently are sold as special solvents (VM&P naphthas, Stoddard solvent, etc.) or are reformed. The extract generally will have an aromatic content of from 55 to 85 percent, depending on the temperature employed and will be of high octane number and of high solvency power. By the addition of another processing step the aromatic content may be increased to over 99 percent. This form of the process has been used to produce pure toluene from petroleum fractions.

A large portion of the installed capacity in Edeleanu units is used for the production of high-quality kerosines from aromatic and high-sulfur crudes. These operations generally are conducted at 10 or 20° F. In this use of the process not only is a kerosine raffinate of excellent color and burning characteristics produced, but the aromatic extracts also frequently are sold at a premium.

The widely increased use of diesel engines has stirred interest in the Edeleanu Process for the production of better diesel fuels. Higher cetane numbers and lower carbon residues and sulfur contents are obtained in SO_2 raffinates produced either from straight-run or from cracked gasoils.

A further indicated use of the process is for the extraction of catalytic cycle stocks. The SO_2 raffinate is substantially as good a cracking stock as the original virgin-gasoil charge. The extract produced is a highly aromatic and refractory material which will crack only to a very low yield of gasoline and is best diverted directly to fuel. The use of the SO_2 extraction step will result in obtaining highest overall gasoline yield with the least use of cracking-plant capacity.



EDELEANU TREATING PROCESS

COURTESY, E. B. BADGER & SONS COMPANY

Ethylene Manufacturing Process

THE LUMMUS COMPANY

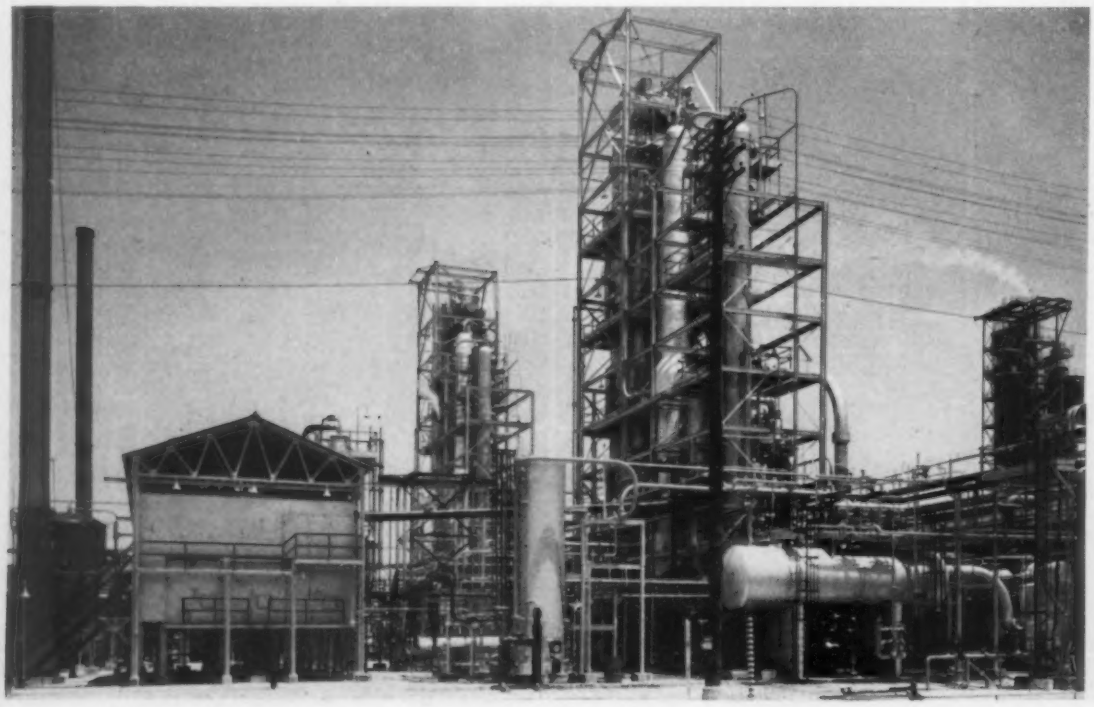
THE great expansion of the petroleum-chemical industry during the past few years has been reflected in the growth of ethylene manufacture and recovery from about 150,000 pounds per day in 1940 to about 600,000 pounds per day in 1947, with more than one million pounds per day scheduled by 1948. While other petroleum source materials, such a benzene, toluene, and the light olefins heavier than ethylene contribute significantly to the tonnage of petroleum chemicals today, ethylene has become probably the most important of these source materials.

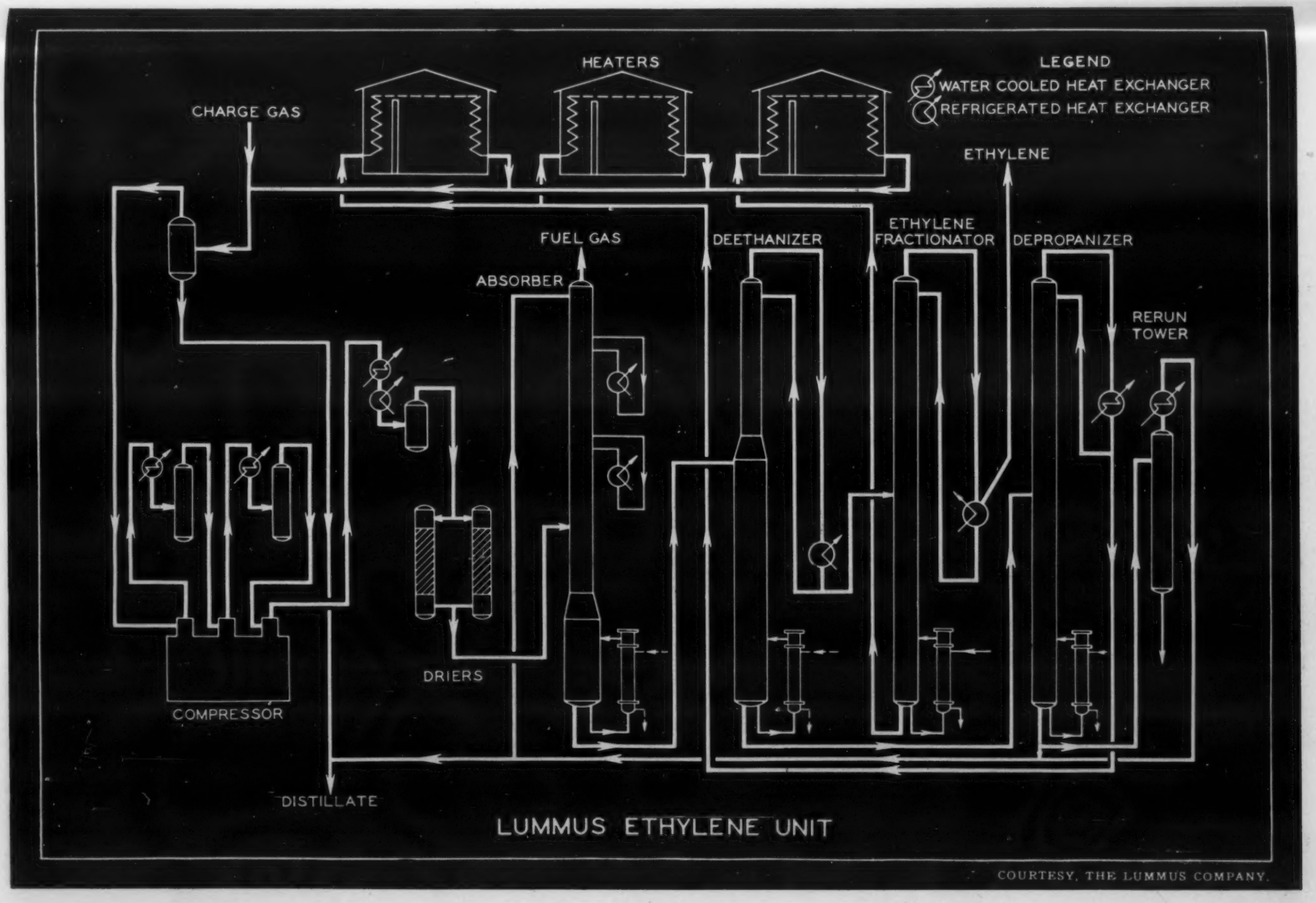
Ethylene may be produced by the pyrolysis of ethane, propane or heavier materials, including reduced crudes, by short-time high-temperature pyrolysis and of course may be recovered from oil-refinery gases where it is the by-product of relatively low-temperature thermal and catalytic-cracking operations. The high-temperature cracking may be conducted in the presence of hot granular refractory or may be accomplished in tubular furnaces. The most important source material for ethylene pyrolysis at the present time is ethane and propane which may be efficiently processed in tubular heaters at temperatures in the range of 1300-1400° F. The flow sheet opposite represents a typical plant designed to produce ethylene by the pyrolysis of ethane, propylene and propane, and to recover ethylene directly from the raw charge gas.

The fractionating absorber is the most important unit in the plant in that it makes the separation between methane and ethylene. It is necessary to avoid loss of ethylene, but on the other hand, retention of too much methane reduces the purity of the ethylene below the desired value since generally no further demethanization is employed. A portion of the aromatic distillate produced in the process is circulated over the absorber at a rate of approximately 3.0-4.0 pounds per pound of feed, at which rate only moderate heat removal in the intercoolers is required in order to recover approximately 99 percent of the ethylene entering in the feed and reject all of the hydrogen and approximately 96 percent of the methane. The rich oil from the absorber flows to the deethanizer where the split is made between ethane and propylene. Inasmuch as both the ethane and the propane-propylene are recycled through the pyrolysis coils, the separation between propylene and ethane need not be sharp, and a low reflux ratio may be used. In this tower it is only necessary to avoid loss of ethylene in the bottoms. The overhead from this tower flows to the ethylene fractionator where the split is made between ethylene and ethane. Recovery of practically 100 percent of the ethylene in the feed to this tower with purities up to 95 percent can be made readily.

Aromatic materials removed from the bottom of the depropanizer are used for absorber lean oil as required, with the balance being delivered to aromatic-distillate production. A small rerun tower is provided for eliminating heavy polymer which builds up in the aromatic distillate.

In this system refrigeration is required in the absorber, the deethanizer and the ethylene fractionator. However, the refrigeration level is from 0° F. upwards and the refrigeration load is therefore moderate amounting to approximately 18 brake-horsepower for 1000 pounds of net ethylene produced per day, exclusive of the charging stock prefractionation requirements. Driers are provided for reducing the water vapor dew point of the absorber charge to less than —40° F., in order to eliminate the formation of ice and hydrates over prolonged periods of operation.





COURTESY, THE LUMMUS COMPANY.

Ethylene Production Process

STONE & WEBSTER ENGINEERING CORPORATION

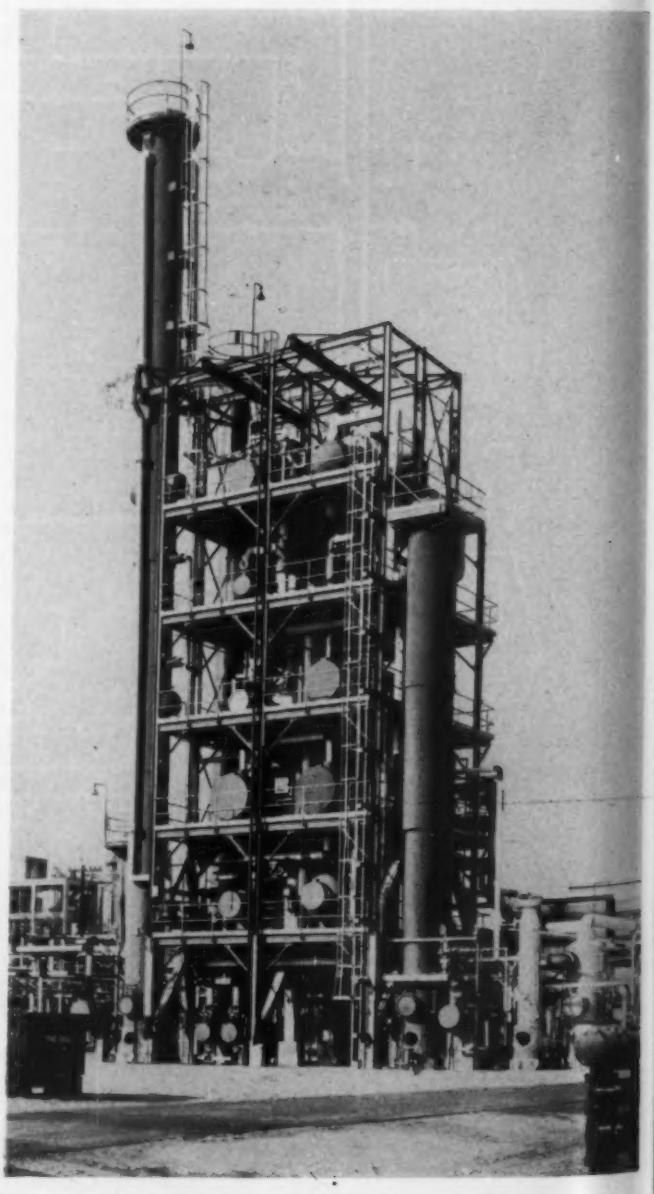
THE ability to produce large quantities of ethylene at low cost has encouraged the rapid increase in production of chemical intermediates which are widely used in the manufacture of alcohols, synthetic rubber, glycols, detergents and a wide variety of plastics and synthetic resins. The two principal sources of ethylene for chemical synthesis are (1) olefin-rich refinery gases and (2) pyrolysis of light hydrocarbon mixtures or crude oil primarily for the production of ethylene or propylene. The cost of producing ethylene by cracking crude oil or distillate stocks is much higher than by cracking an ethane-propane mixture or a refinery depropanizer overhead stream. Hence, most large ethylene producers are located near a cheap source of refinery gas or light hydrocarbons.

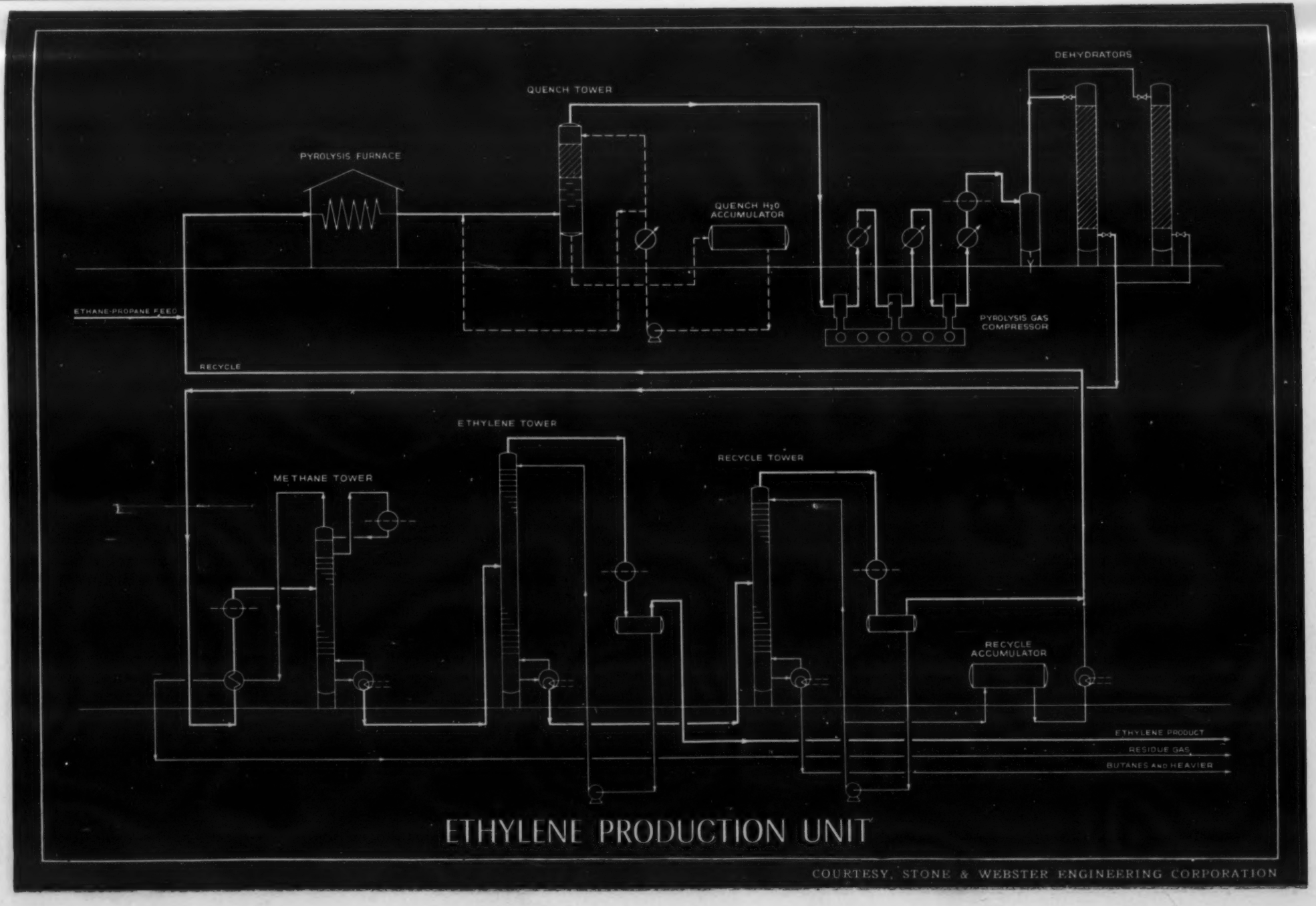
Most of the chemical synthesis processes require that the ethylene concentration be greater than 90 percent and that the product gas be free of propylene and higher-molecular-weight hydrocarbons. The separation of the ethylene from the cracked-gas mixture and its subsequent purification usually is accomplished by fractional distillation at subatmospheric temperatures.

The process shown on the accompanying flow sheet, presented through the courtesy of Stone & Webster Engineering Corporation, illustrates a typical ethylene production unit based on cracking an ethane-propane mixture.

The fresh feed, together with recycled ethane and C_3's, is charged to the pyrolysis furnace and heated to 1450-1500° F., where optimum conversion to ethylene takes place. The coil-outlet gases are water quenched and compressed in three or four stages to 600 psig. The gas from the last stage of compression is cooled with water, then subcooled with refrigerant to approximately 70° F. to remove the maximum amount of water from the compressed gas stream before introducing it into the dehydrators. Drying of the gas to a water dew point of approximately minus 100° F. is effected by passing the gas through a bed of activated bauxite or alumina. The dried gas is cooled by heat exchange and refrigeration and introduced into the methane tower where hydrogen and methane are taken overhead. Reflux is produced in a run-back type condenser refrigerated by the evaporation of ethylene. The reflux temperature is approximately minus 130° F. The bottoms from the methane tower flow to an ethylene tower where high-purity ethylene is removed as an overhead product. Reflux is produced at 0 to minus 15° F. in a condenser refrigerated with propane or ammonia.

The subsequent processing of the ethane and heavier components which comprise the ethylene-tower bottoms depends upon the number and purity of products required. An ethane tower and a propane tower are required if a propylene-propane product is desired. If ethylene is the only primary product, the ethane and C_3's may be recycled to the pyrolysis furnace, in which case, a single tower would suffice to separate the recycle stream from the C_4's and heavier components.





ETHYLENE PRODUCTION UNIT

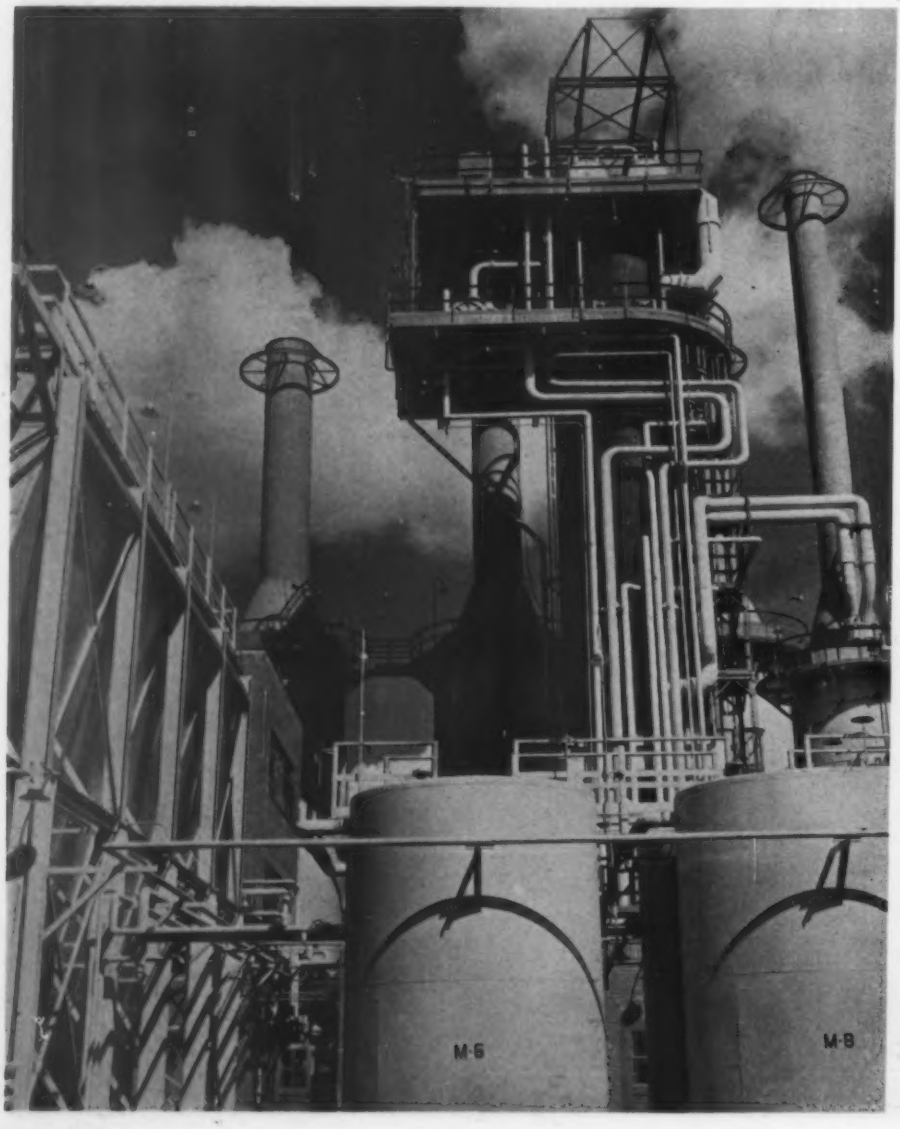
COURTESY, STONE & WEBSTER ENGINEERING CORPORATION

Filtrol Fractionation Process

FILTROL CORPORATION

FILTROL Fractionation is the name of the process in which Filtrol, the highly activated adsorbent made by Filtrol Corporation, is injected into a hydrocarbon oil stream as it flows to fractional distillation equipment. The purpose of this relatively new technique is to combine fractional distillation and continuous filtration into a single and more economical process. The process is applicable to the refining of solvent-treated stocks, long residuals, low-asphaltic or treated lube crudes, and dewaxed cylinder stock solutions. By combining distillation and decolorization, substantial savings in equipment, upkeep, maintenance and operating costs are effected.

On the opposite page is a flow diagram of a Filtrol Fractionation plant presented through the courtesy of Filtrol Corporation, 634 South Spring Street, Los Angeles, California. As indicated in the flow sheet, a portion of the charge to the pipe heater is continuously by-passed through a small mix tank. This tank is equipped with a mechanical agitator which mixes the Filtrol with the oil, thus forming a readily pumped oil-adsorbent slurry. The Filtrol is automatically fed to this tank by a gravimetric feeder, insuring accurate control of the adsorbent feed at all times. The feeder is supplied with Filtrol by a scroll-type conveyor from the overhead storage bin. The by-passed oil containing the Filtrol rejoins the main flow of the charge and continues through the heat exchangers before entering the pipe heater. The temperature of the heater outlet is instrument controlled. The hot oil-adsorbent slurry then enters the vaporizer section of the vacuum fractionating column where the unvaporized portion which contains all the Filtrol passes over a number of stripping trays before falling into the soaking section. The lighter cuts thus receive an adsorbent treatment in passing through the exchangers and furnace, after which the adsorbent charge is concentrated in the bottoms, which require heavier treatment. The vaporized material is fractionated into the desired cuts, each of which passes through a steam stripper which eliminates undesirable odor and insures color-stable products.



The bottoms and Filtrol in the soaking section of the tower are agitated and stripped by the injection of steam into the bottom of the vessel. The soaking time or contact time is regulated by controlling the liquid-level in this section. The slurry then passes through the bottoms exchangers and into an insulated surge tank from which a continuous-vacuum rotary filter is fed. The spent adsorbent is filtered from the oil which passes through a cooler to finished oil storage. The spent cake is continuously removed by a screw conveyor.

In certain instances, the Filtrol Fractionation Process has definite benefits over contact filtration finishing. The refiner's position, process- and market-wise, must be carefully analyzed to determine the extent of these advantages for each specific case. Important advantages realized by the use of this refining technique include reduced operating cost, lowered construction cost, and maximum adsorbent economy.

COURTESY, FILTROL CORPORATION

Fluid Catalytic Cracking Process

THE M. W. KELLOGG COMPANY

THE primary purpose of the Fluid Process as with other petroleum cracking operations is to break down long-chain and high-molecular-weight hydrocarbons into molecules boiling within the gasoline range. The Fluid Process differs from other catalytic-cracking processes in that the reaction is effected by a *powdered* catalyst which is circulated essentially as a fluid with the oil to be cracked. Advantages of the system as a means of controlling and handling catalyst are:

(1) A highly turbulent bed in the reactor provides intimate contact between catalyst and vapor.

(2) The ability to control reaction conditions closely over a wide range produces optimum yields with uniform quality of products.

(3) Continuous uniform regeneration is obtained with minimum degradation of catalyst activity.

(4) The fluid catalyst may be circulated freely without regard to limitations of mechanical transfer equipment. Flows are maintained by self-perpetuating hydrostatic heads.

(5) Heat is conveyed from the regeneration system to the reaction system without the danger of thermal cracking and coking.

Hot catalyst at a temperature of approximately 1100° F. passes from the regenerator into the regenerator standpipe down which it flows to the oil-injection point where it flash vaporizes the fresh-oil charge which has been pre-heated to balance the total heat requirements of the reaction.

The mixture of oil vapors and catalyst is forced up the reactor-feed line by the pressure head maintained in the regenerator plus the force of gravity exerted on the dense catalyst in the catalyst standpipe, the pump serving merely to bring the oil to the oil-injection point.

The hot mixture enters the reactor where the catalyst settles to a definite level and forms a "bed," depth of which regulates the time of reaction, and can be varied at will. The bed is maintained in a fluid, turbulent condition by the entering feed vapors. Continually passing upward, these vapors effect intimate contact of oil with catalyst and produce a uniform temperature (900° - 950° F.) throughout the bed. As cracking progresses, coke forms on the catalyst.

The spent catalyst, laden with coke, is continually and automatically withdrawn through a slide valve to the spent-catalyst stripper where the adsorbed and entrained vapors are dissociated from the catalyst and carried by a counter-current stripping medium back into the top of the reactor vapor space. Spent catalyst separates from the oil vapors by gravity, drops into the spent-catalyst standpipe and through an automatically controlled slide valve to the air-injection point where its density is reduced by a current of air which "fluffs up" the catalyst so that it flows as a fluid up the regenerator return line.

The oxygen in the air admitted for "fluffing up" purposes continuously regenerates the catalyst by burning off coke. This action brings the catalyst to a temperature of about 1100° F. and makes it an effective heat-transfer medium for vaporizing the fresh-oil feed and supplying the heat necessary for the cracking reaction. The reactivated hot catalyst is retained in the regenerator and is constantly withdrawn into the standpipe for re-use as needed.

The combustion gases pass through cyclone separators in the top of the regenerator where they follow a high-velocity spiral path which throws the heavier catalyst particles to the outside of the separator by centrifugal force. The recovered catalyst is returned to the bed while the flue gases enter the regenerator exit line and pass through the flue-gas cooler where their contained heat is used to generate high-pressure steam by heat exchange with water.

The cooled flue gases then pass to the electrical precipitator. Flue gases are vented to the atmosphere. Recovered catalyst passes from the precipitator into the recovered-catalyst line and is blown back to the regenerator.

The combined hydrocarbon gases from the spent-catalyst stripper and the reactor join in the vapor space above the catalyst bed and enter cyclone separators where catalyst is recovered and returned to the main bed. The cracked gases pass through the reactor overhead line to the fractionator scrubbing section where the small quantity of residual catalyst fines is diverted to the bottom of the fractionator and passes with the heaviest oil to the slurry settler, an integral part of the fractionator in which the catalyst settles out by gravity. The clarified decanted oil is withdrawn from the top of the settler for use as fuel oil or for further processing.

The thickened catalyst slurry passes into the catalyst-return line and is returned to the reactor. This completes the circuit of catalyst in the system.

The vapor stream entering the fractionator passes upward from the scrubbing section through the bubble plates of the fractionator and usually is separated into wet gases, unstabilized gasoline, light gasoil, and heavy gasoil.

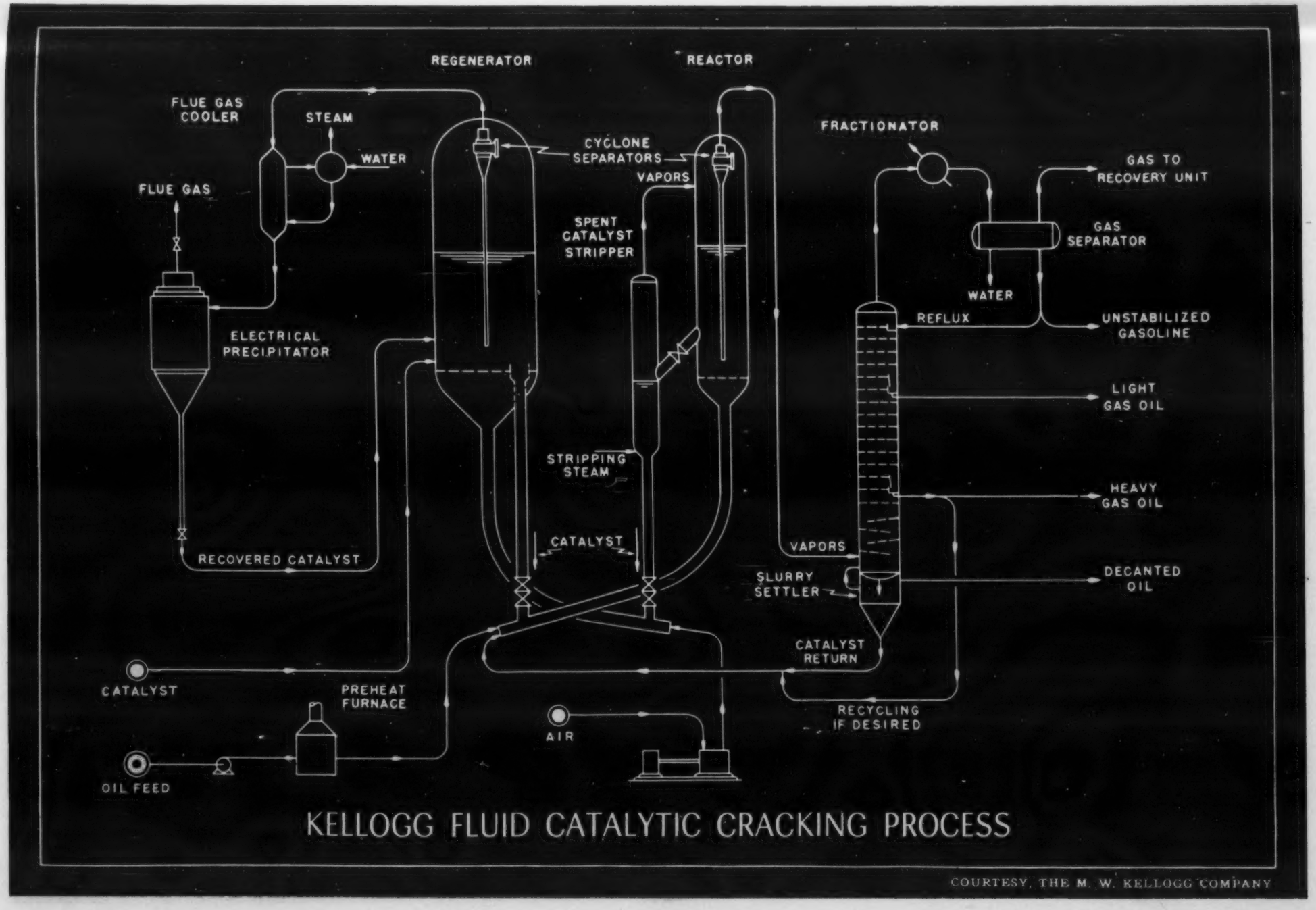
The heavy gasoil, or the entire fractionator side stream, can be diverted for a recycling operation to the recycle-oil line and so returned to the reactor for further cracking with the fresh feed.

The table shows results obtained from single-pass operation at 50 percent conversion level on five representative feed stocks.

Comparison of Single Pass Yields at 50 Percent Conversion Gasoils from Representative Crude Sources. Synthetic Catalyst. 900° F.

	West Texas	Coastal	Mid-Cont.	East Venez.	Arab.
Product Inspections—					
Propylene in Propanes—Vol. Percent	73	70	75	70	75
Butane-Butene Fraction:					
Butenes—Vol. Percent	58	55	58	48	60
i-Butane in Sats.—Vol. Percent	80	80	83	83	83
Debutanized Motor Gasoline:					
Reid Vapor Pressure—lb.	6.1	6.2	6.3	6.5	7.0
Gravity, °API	55.1	55.0	56.9	56.0	57.0
ASTM Percent (D plus L) at 158 °F	27	27	28	27	28
212	50	50	51	49	54
284	68	70	73	70	70
356	88	90	91	90	90
Octane Numbers—					
ASTM—Clear	81.1	82.0	82.0	80.5	82.0
CFRR—Clear	94.6	95.0	95.1	94.2	96.0
Total Cycle Oil:					
Gravity, °API	24.5	23.0	26.8	25.0	26.0
ASTM 50 Percent Pt. °F.	600	610	604	580	555

Licenses for this process are obtainable from The M. W. Kellogg Company.



KELLOGG FLUID CATALYTIC CRACKING PROCESS

COURTESY, THE M. W. KELLOGG COMPANY

Fluid Catalytic Cracking Process

STANDARD OIL DEVELOPMENT COMPANY

FLUID catalytic cracking, which was the source of a substantial part of the military petroleum products used in World War II, is operating to produce peacetime products of a wide variety. It, also, is being adapted for conversion of natural gas into liquid products, where it represents an advancement over the Fischer-Tropsch process.

In the Fluid process, finely divided catalyst is suspended in gas or vapor so it flows through the equipment in a form resembling a liquid in many respects. Thus it circulates continuously between reaction and regeneration zones, with the heat from the regenerator being transferred to the reactor, thereby replacing the need for cooling surface in the regenerator and reducing the preheat requirement for the feed. Since it requires no furnace to vaporize the feed, coking difficulties do not exist.

Pressure for catalyst circulation is provided by a standpipe, in which a column of catalyst is maintained in a fluidized state by addition of aeration gas, so controlled that the column builds up pressure at the base equal to the weight of solids above, consequently no mechanical circulation is required. By operating the vessels at relatively low velocity, a bed of dense solids is formed due to their tendency to settle out, whereby it is possible to obtain the necessary catalyst holdup in vessels of moderate size.

With reference to the accompanying flow diagram, oil feed is injected into the hot regenerated catalyst, which supplies the required heat, the catalyst rate being regulated to maintain required reactor temperature. This catalyst-vapor mixture flows into the reactor, where the catalyst begins to settle out, forming a dense bed, which resembles a violently boiling liquid with a rather clearly defined level. Vapors leave the top of the reactor through separators which return most of the entrained catalyst to the bed.

Cracked products then are processed in conventional fractionating equipment, in which any remaining catalyst is concentrated for return to the regenerator.

In the reaction step a carbonaceous deposit accumulates on the catalyst, which is removed from the reactor through a stripping zone where the hydrocarbon vapors are removed. Spent catalyst is then suspended in air and carried into the regenerator where the coke is removed by combustion. Heat thus released is absorbed by the circulating catalyst. Flue gases leave the regenerator through cyclones, are cooled in conventional waste-heat boilers, then flow through an electric precipitator where the dust content is reduced to a low value before the gases are vented.

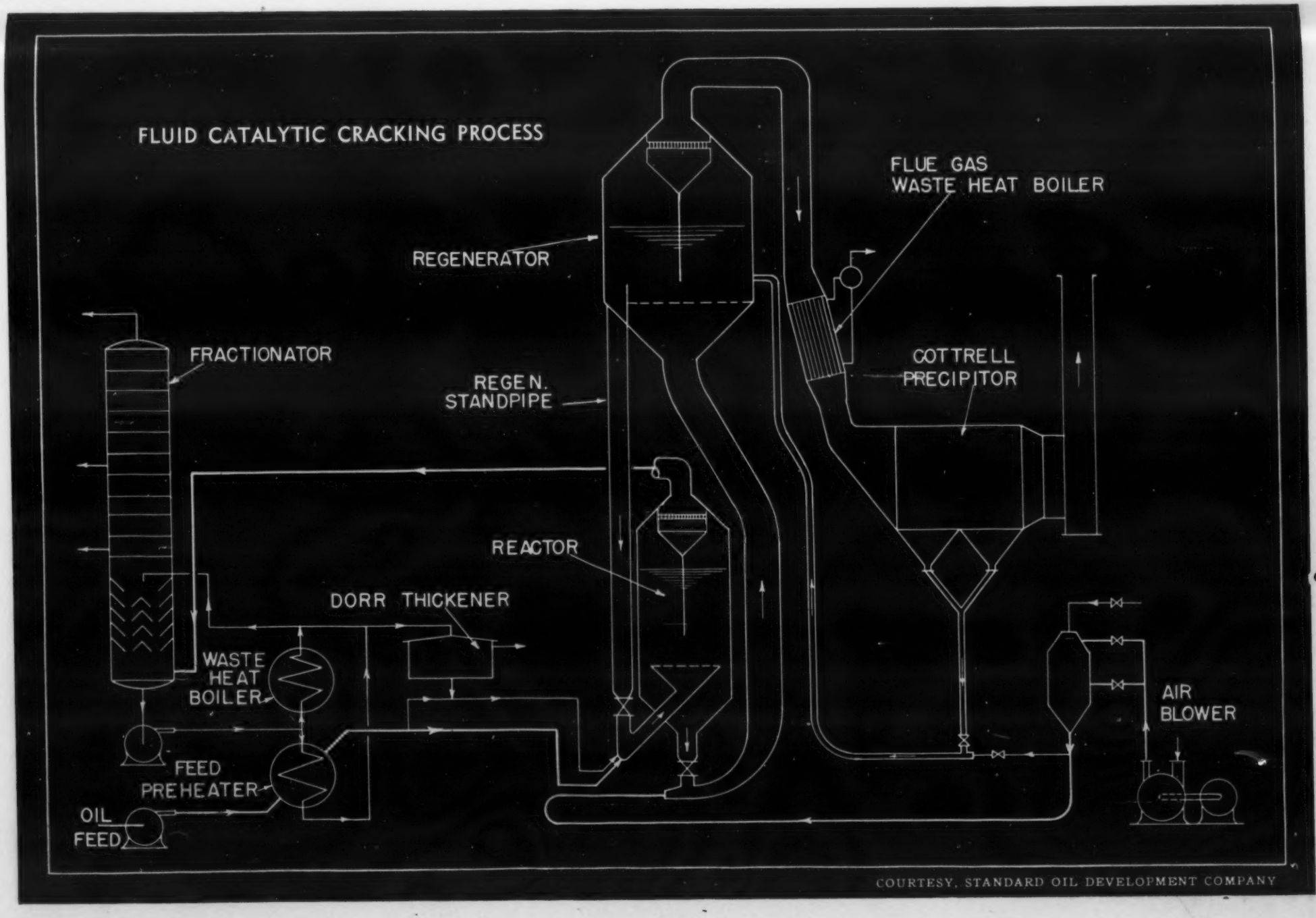
High yields of motor gasoline are obtained with clear octane numbers of 81-83 ASTM and 92-99 CRF. By-products such as isobutane and light olefins for alkylation, polymerization or for synthetic-rubber ingredients are recovered.

Feed stocks in commercial operation range from naphtha cuts to heavy gasoils and deasphalted residuums, while recycling operations have been found attractive in some instances. Large-scale tests have been conducted on feeding whole reduced crude. Flexibility of the process has been demonstrated through uninterrupted runs of more than a year, and by general high-service-factor levels. High catalyst recovery has been established in commercial plants, based on a circulation rate corresponding to a loss averaging about 0.25 pounds per barrel of feed.

Fluid process units now operating or under construction in this country represent an operating capacity of more than 1,000,000 barrels per stream day, or more than half the total domestic catalytic cracking capacity. Capacity of the units ranges from 2,000 to 30,000 barrels per stream day.

Adaptation of the Fluid process in natural gas conversion has been explained by Robert P. Russell, president of Standard Oil Development Company, as applying to the conversion of the synthesis gas into liquids where a cheap iron-type catalyst is used. Cooling tubes immersed directly into the fluid bed are able to extract the heat of reaction with high temperature differences and good heat-transfer coefficients so that the heat transfer area is reduced to less than 5 percent of the requirements in the conventional Fisher-Tropsch process. At the same time the extremely turbulent conditions in the fluid bed maintain a surprising degree of uniformity in bed temperatures. Approximately 80 percent of the hydrocarbons produced after polymerization of C_3 and C_4 olefins represent gasoline of high quality, requiring only bauxite treating for finishing, while the remainder of the hydrocarbon product is of diesel-oil quality.

Information for this description and the flow diagram were provided by Standard Oil Development Company, 30 Rockefeller Plaza, New York 20, N. Y.


FLUID CATALYTIC CRACKING PROCESS
FLUE GAS
WASTE HEAT BOILER
REGENERATOR
FRACTIONATOR
REGEN.
STANDPIPE
COTTRELL
PRECIPITOR
REACTOR
DORR THICKENER
WASTE
HEAT
BOILER
AIR
BLOWER
FEED
PREHEATER
OIL
FEED
COURTESY, STANDARD OIL DEVELOPMENT COMPANY

Furfural Refining Process

TEXACO DEVELOPMENT CORPORATION

FURFURAL is an extractive solvent extensively employed for refining a wide range of lubricating oils of any viscosity and diesel-fuel oil. Furfural originally was chosen as most suitable for solvent extraction of lubricating oils because of its high degree of selectivity for the undesirable components of petroleum oils without possessing a high solvent power for the desirable components. It is unique in this respect. The addition of water or an "opposing" solvent is not required to control this solvent power. Furfural also was chosen because of its stability, low cost, availability and desirable physical characteristics. Being non-acidic and non-toxic, furfural can be handled without special protective provisions. Its boiling point is sufficiently low (323° F.) to permit its recovery from the lowest-boiling-point lubricating-oil stocks and even from diesel-fuel oil or furnace oils without appreciable loss of oil or solvent. Its low solid point (—38° F.) eliminates the danger or solidification in lines or tanks in cold weather. The high specific gravity of furfural insures rapid and complete separation at normal operating temperatures in extraction towers of relatively small diameter. The critical solution temperature of furfural is high enough to permit its application to waxy or viscous oils at temperatures that insure treatment in a non-viscous, completely liquid state. The stability and recoverability of furfural is emphasized by the low operating losses experienced by commercial refining units.

The wide application of furfural to solvent refining is illustrated by the fact that units ranging from 60 to 6000 barrels per day of charge oil capacity, totaling over 41,000 barrels per day, have been installed in some 20 refineries for the solvent extraction of naphthenic or paraffinic distillate and residual oils. Additional furfural-extraction units having a combined charge capacity of over 19,000 barrels per day are under contract for construction.

The furfural-extraction unit represented by the accompanying flow diagram comprises an extraction or treating section, a raffinate-recovery section, an extract-recovery section, water-removal facilities, process surge tanks and pumping equipment. Furfural, however, because of its stability over a wide range of operating conditions, lends itself to a variety of methods of recovery from raffinate and extract solutions. Different types of the solvent-recovery systems have been developed and used, in order to provide for maximum operating economy of each particular installation.

One of the major improvements in the process in recent years has been the development of a highly efficient treating tower, in which an increased degree of fractionation is obtained between the relatively insoluble, desirable lubricating-oil fractions and the more soluble, undesirable fractions. Untreated oil is introduced into the continuous countercurrent extractor at some mid-point and at a predetermined temperature of from 110° to 200° F. The temperature of the furfural charged near the top of the extractor varies depending on the charge stock, up to 290° F. or higher. A substantial temperature gradient is maintained between the top and bottom of the tower by indirect water coolers provided to control the temperature spread.

The extraction tower generally is operated with a prevailing oil phase through which the dispersed furfural phases passes downward over Raschig ring nests which afford an extensive surface of contact between the phases. This, in turn, promotes rapid and relatively complete equilibrium by interchange between the phases without mechanical mixing or pumping equipment. The concentration of undesirable oily constituents in the furfural phase increases toward the bottom of the tower from which the extract solution is withdrawn continuously. Conversely, the oil quality improves toward the top of the tower from which practically all oil of predetermined raffinate quality that is present in the charge stock, overflows with a small percentage of furfural dissolved in this phase.

Further improvement in fractionation between desirable and undesirable constituents has been secured by the controlled recirculation or "refluxing" of extract to the lower portion of the tower. The recirculated extract functions in much the same way as reflux to a distillation column, in that it sharply fractionates oil from the extract solution, and this oil rises again to the refining zone for re-extraction and consequent improvement in raffinate yield.

The recovery of solvent is accomplished by continuous distillation of furfural from the raffinate and extract phases in the respective recovery sections followed by steam stripping of each product. The overhead condensate from the steam strippers separates into two furfural-water layers, one rich and the other lean in furfural. The recovery of furfural from water is accomplished in a simple continuous fractionating system in which dry furfural is removed from the bottom of one tower and water from the bottom of another tower, taking overhead from each a constant-boiling mixture of water and furfural which is condensed and returned for redistillation.

This discussion is presented through the cooperation of Texaco Development Corporation, 135 East 42nd Street, New York, New York, licensors of the Furfural Refining Process.

FURFURAL REFINING UNIT
COURTESY, TEXACO DEVELOPMENT CORPORATION
WET FURFURAL VAPOR CONDENSER
SETTLING DRUM
FURFURAL-FROM-WATER STEAM STRIPPER
EXTRACT MIX HEATER
REFINED OIL MIX HEATER
HOT FURFURAL ACCUMULATOR
VAPOR HEAT EXCHANGER
SOLVENT-EXTRACT MIX EXCHANGER
WATER-FROM-FURFURAL STRIPPER
EXTRACT PRESSURE FLASH TOWER
EXTRACT ATMOSPHERIC FLASH TOWER
REFINED OIL STEAM STRIPPER
EXTRACT STEAM STRIPPER
EXTRACT COOLER
LOW PRESSURE REFINED OIL FLASH TOWER
REFINED OIL-REFINED OIL MIX EXCHANGER
REFINED OIL COOLER
FURFURAL COOLER
CHARGE OIL HEATER
EXTRACT MIX INTERCOOLERS
TREATING TOWER
EXTRACT MIX EXCHANGER
FURFURAL STORAGE
EXTRACT STORAGE
FURFURAL REFINED OIL STORAGE
CHARGE OIL STORAGE
LEGEND

Gas-Concentration and Stabilization Process

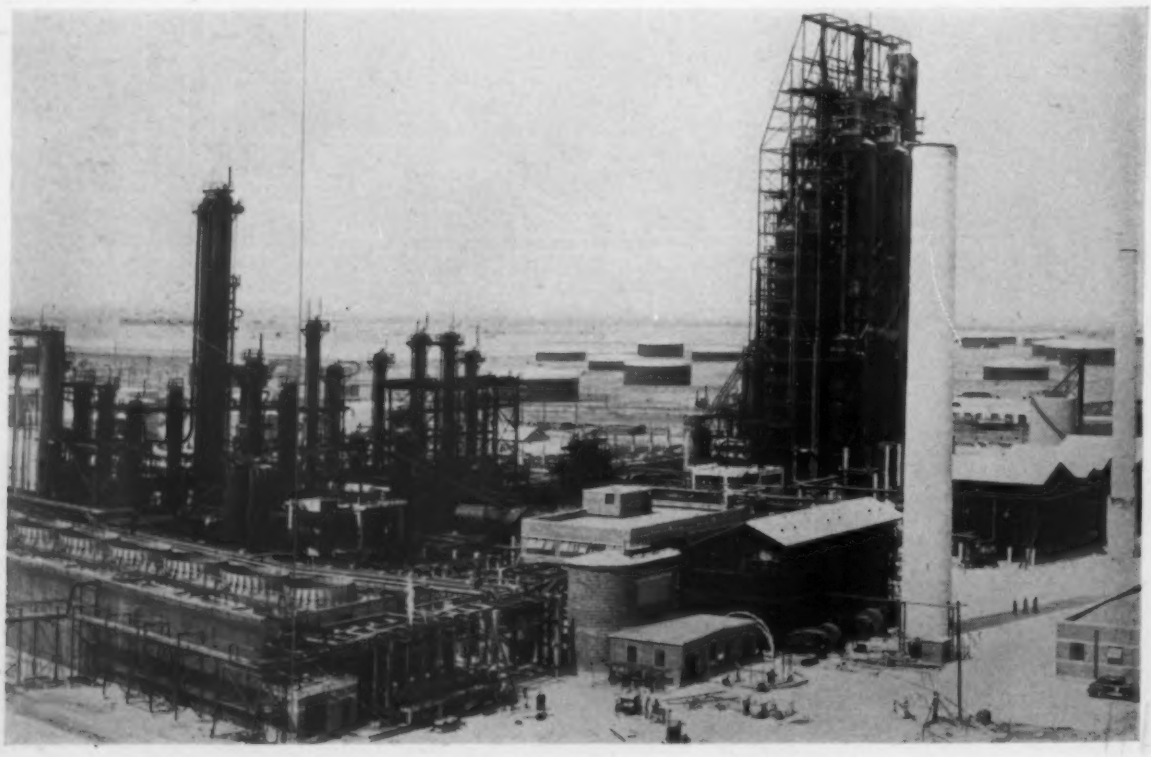
THE LUMMUS COMPANY

GAS-CONCENTRATION and stabilization plants are among the earliest secondary processes used in petroleum refining. The necessity for such plants dates back to the earliest days of thermal cracking, especially in situations where low-pressure separation of the cracking-plant effluent was conducted. As the art of thermal cracking developed there was a trend towards employing high-pressure-fractionation systems and thereby minimize the necessity for gas-recovery systems.

Practically all refineries in operation today include some form of gas-recovery and stabilization plant. The design of this plant depends to a considerable extent upon the type of processing units employed and the amounts of wet gas and unstable distillates to be handled. However, there have been significant advances in methods of handling these materials, with the result that substantial savings in both investment and operating costs can be made over antiquated or obsolete systems. Each refinery situation requires special consideration.

The advent of catalytic cracking brought about a great increase in the installation of gas-concentration and stabilization facilities for the twofold reason that catalytic-cracking plants produce large quantities of pentane and lighter materials and that these plants must of necessity fractionate the reactor effluent at low pressure. The most suitable type of gas-concentration facilities for operation in conjunction with catalytic cracking will depend to some extent upon the type of catalytic-cracking operation and the nature of the charging stock. Gas-concentration units of a variety of designs, however, may be used for accomplishing any required separation and recovery. The Lummus Company has given careful consideration to the many possible types of plant and has determined that the system shown in the flow sheet opposite represents one of the simplest and most economical for situations where it is necessary to recover all C_4's and high recovery of C_3's.

This plant consists of a compression system for raising the pressure of the wet gas to approximately 150 psig and contacting this material with the unstable distillate; an absorber for processing the wet gas from the contact drum with debutanized gasoline and heavy naphtha; and a debutanizer for producing debutanized pasoline and an overhead C_3, C_4 condensate stream. The debutanizer in effect acts as a still for the absorption naphtha, however part of the lean oil flows directly from the low-pressure-fractionating system of the catalytic-cracking unit to the top of the absorber for the purpose of sponging the small amount of pentanes and heavier material evaporated from the debutanized gasoline making up the bulk of the lean oil. The debutanizer overhead condensate may be charged through a Girbotol unit to a catalytic polymerization plant or may be charged to a depropanized for recovery of butane-butylenes and an overhead C_3 condensate. The debutanizer may be reboiled with 450-pound steam or with hot circulating oil from the low-pressure-fractionating system, the latter usually being more desirable. In some situations it has been found desirable to provide a fractionating absorber equipped with a reboiler in order to reduce the gas recycle from the debutanizer and the amount of gasoline recycled to the absorber for lean oil.



LUMMUS GAS CONCENTRATION UNIT
WET GAS & UNSTABLE GASOLINE FROM CATALYTIC CRACKING UNIT
COMPRESSOR
ABSORBER
DEBUTANIZER
DEPROPANIZER
150# STEAM
450# STEAM
FUEL GAS
HEAVY NAPHTHA FROM CATALYTIC CRACKING UNIT
DEBUTANIZED GASOLINE
GIRBOTOL PLANT
BUTANES
CONDENSATE
OFF GAS
COURTESY, THE LUMMUS COMPANY.

Girbotol Process

THE GIRDLER CORPORATION

THE Girbotol Process (patented) for the separation of acidic constituents from gaseous and liquid mixtures, is widely used for purification of gases and liquids in the petroleum industry. The process is employed for removal and recovery of hydrogen sulfide from natural and refinery gases, for simultaneous desulfurization and dehydration of natural gas, for purification of liquid hydrocarbons, and for separation of carbon dioxide from hydrogen used for hydrogenation of oil and synthesis of iso-octane.

Operation of the Girbotol Process is based upon the discovery that aliphatic amines have a strong affinity for acid gases such as hydrogen sulphide and carbon dioxide at or near atmospheric temperature, but at higher temperatures the affinity drops off rapidly and the gas is expelled. The process operates in a continuous cycle, absorbing acid gas from a gas stream at atmospheric temperature and expelling it at another point by raising the temperature of the absorbent. Because the amines are strong bases, freely soluble in water, large volumes of acid gas may be removed per volume of solution circulated. The efficiency of the process is therefore high. Hydrogen sulphide is recovered in concentrated form.

The cycle and equipment employed in the process are almost identical with those of a gasoline absorption plant (see flow chart). Gas to be treated flows up through a bubble tower or absorber. Amine solution, at atmospheric temperature, enters the absorber and flows down the tower from tray to tray picking up the acid gas. Purified gas leaves the absorber at the top.

Amine solution, saturated with acid gas, passes through heat exchangers, where its temperature is raised to 190-200° F., and into the upper part of the reactivator, which also consists of a bubble tower, containing in its base a tubular reboiler section and at the top a reflux condenser. The amine solution, flowing down the tower, is heated to 215-220° F. by steam rising from the boiling solution in the reboiler section, and the acid gas is expelled and flushed out by ascending vapors. The acid gas leaves the reactivator saturated with water vapor at 190-200° F. and flows through the condenser where it is cooled by water and the steam condensed. The condensate is returned to the reactivator. Hot, lean solution, stripped of acid gas flows from the base of the reactivator to a pump, thence through the exchangers in counter current relation to the cold, rich solution. From the exchangers the solution passes through water coolers, and finally back to the top of the absorber. The plant is entirely automatic in operation, requiring only casual supervision.

Desulphurization — Separation of hydrogen sulphide from hydrocarbon gases is accomplished as described above. The gases may be treated at high or low pressure with equal success, but gas under pressure is purified more economically because smaller towers are required and larger volumes of hydrogen sulphide are removed per gallon of solution circulated. The process is well adapted for purifying gas containing a high percentage of hydrogen sulphide. The rate of reaction in the absorption stage is so rapid that complete stripping may be obtained in a single tower. Because an aqueous absorbing solution is employed, practically no hydrocarbon constituents are removed from the gas if the physical conditions of temperature and pressure are controlled. This makes it possible to apply the process for the purification of wet gas before the extraction of gasoline and results in lower subsequent treating costs to purify the gasoline.

The Girbotol Process is useful for the purification of gaseous feed to a polymerization plant. Unless the poly feed is substantially free of hydrogen sulphide, mercaptans and other sulfur compounds formed during polymerization must be removed from the poly product. Such after-treatment is expensive and damaging to the octane number and the blending value of the poly product. The poly feed may be purified with the Girbotol Process at any temperatures up to 180° F.

Dehydration and Desulphurization—In several Girbotol plants purifying natural gas for pipe line distribution, simultaneous dehydration of the gas is accomplished by employing a dehydrating liquid such as diethylene glycol in solution with the Girbotol absorbent. H_2S and water are both scrubbed from the gas, and expelled together from the Girbotol reactivator, the H_2S assisting in carrying the excess water from the tower.

It is thus possible to sweeten sour natural gas and dry it in a single, simple plant. Such gas is being distributed through pipe lines to domestic and industrial customers, without troubles from pipe line freeze-ups.

Desulphurization of Liquid Hydrocarbons—The removal of hydrogen sulphide from liquid hydrocarbons, such as propane, butane, and gasoline, is being carried out successfully in a number of Girbotol plants. Packed towers are used for contacting the liquid streams, while the amine solutions are reactivated in the usual manner.

Carbon Dioxide Removal from Hydrogen—The Hygirtol Process for the production of high-purity hydrogen from hydrocarbons and steam utilizes the Girbotol process for the removal of carbon dioxide from hydrogen. The Girbotol process is the only cyclic process in commercial use for effecting complete removal of carbon dioxide from other gases at or near atmospheric pressure.

This information is presented through cooperation of The Girdler Corporation, Louisville, Kentucky, owner and licensor of the Girbotol Process.

COURTESY, THE GIRDLER CORPORATION

Gray Catalytic Desulfurization Process

TEXACO DEVELOPMENT CORPORATION

THE Gray catalytic desulfurization process, now commercially used for the elimination of both mercaptan and non-mercaptan sulfur compounds from lighter petroleum fractions, meets the need for a catalytic process utilizing a low-cost catalyst and enabling the employment of a highly selective procedure to minimize product losses through undesirable side effects.

To accomplish the desulfurization, gasoline vapors are passed through a bed of the catalyst at an elevated temperature, the temperature dependent on the type of charge stock and the degree of effect desired. The pressure of operation, while usually of a low range, is sufficient to secure the desired flow of vapors through the process and auxiliary equipment.

The catalyst used is of the solid adsorbent type, such as fuller's earth and the like, which is low in cost and readily available from many widely spaced deposits in the United States, and therefore conveniently accessible to all refining centers in this country. It has been found that a wide selection of effective clays can be used and several plants operating the Gray catalytic desulfurization process are using different fuller's earth, choice of clay being made after consideration of operating and economic factors. A flow diagram is shown.

In the application of the process to straight-run gasoline (referring to the flow diagram), the desired fraction is heated to a temperature in the order of 700° F. in a furnace of simple design. The heated vapors pass through a bed of solid adsorbent catalyst at a flow rate dependent upon the character of the feed and nature of the sulfur compounds, wherein the conversion of mercaptan and non-mercaptan sulfur compounds takes place. The vapors leaving the catalyst chamber pass in heat exchange with the incoming gasoline and go then to a condenser and separator. A large part of the sulfur present in the original mercaptan and non-mercaptan sulfur compounds is converted to hydrogen sulfide, which is partially removed in the separator and the remainder removed by means of either a caustic wash or simple stabilization. One of the important economic factors realized as a result of application of this Gray process to straight-run gasoline is that, other than removal of hydrogen sulfide, no further treatment of the gasoline is required.

The desulfurization of 100% cracked gasoline ordinarily requires that the gasoline be pre-treated by the conventional Gray treating process prior to desulfurization in order that the most economical catalyst life be realized. Catalyst yields here range upward of 1,200 bbls./ton. The use of the conventional Gray process prior to desulfurization can be additionally justified on the basis that it effects refining of the gasoline by the removal of diolefins and thereby obviates the need for using inhibitors in the final product.

Untreated cracked gasoline is being desulfurized in a commercial installation with substantial lead savings in the form of a 50-50 blend with straight-run gasoline. In this case the straight-run gasoline, as well as the cracked, has poor lead susceptibility, and both cracked and straight-run components are together improved by desulfurization treatment.

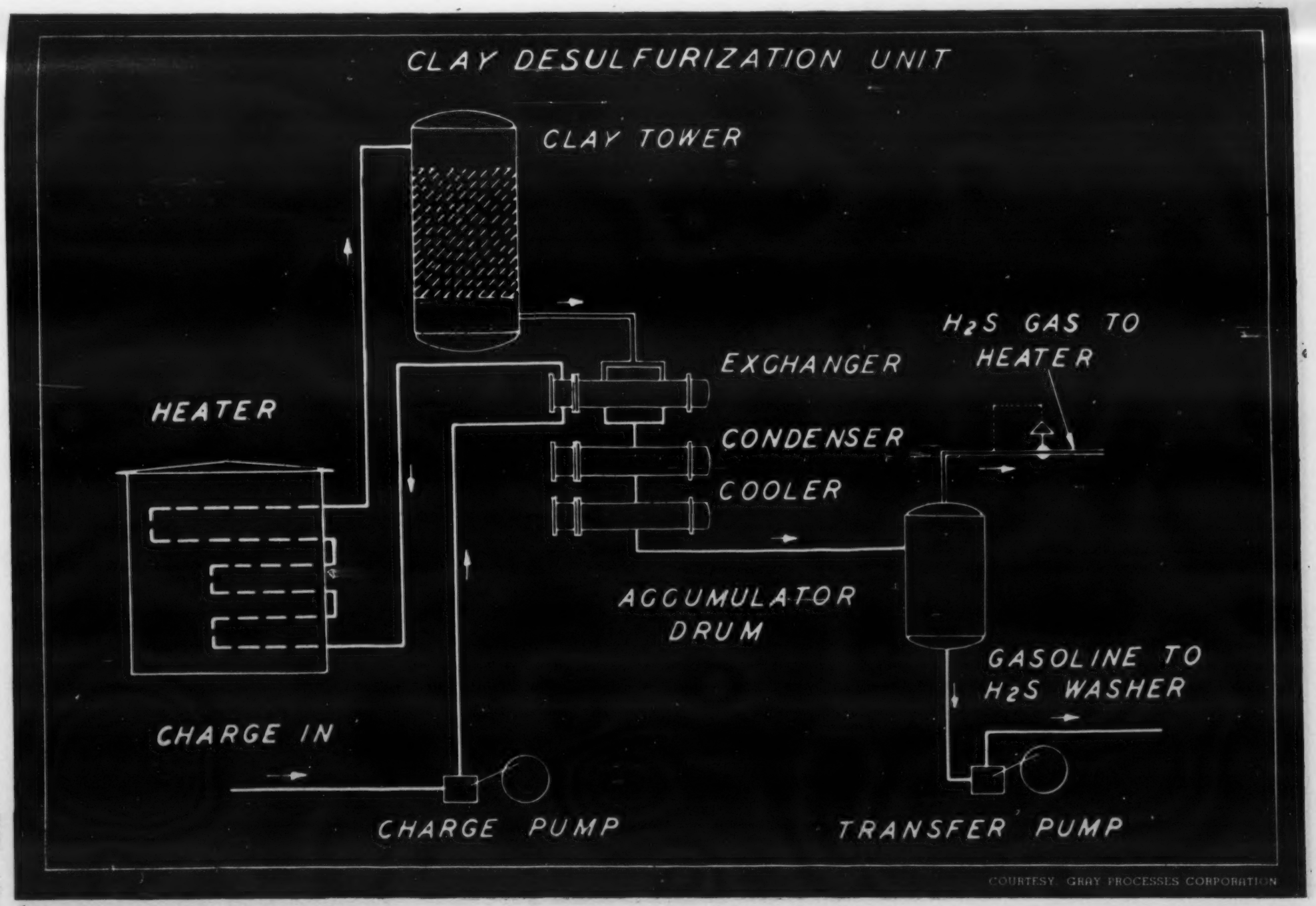
Catalyst yields range from 800 to 1000 barrels per ton for this operation and the low cost of the fuller's earth type catalyst used in the Gray desulfurization process makes it possible to realize substantial net savings over the cost of operation at these catalyst yields.

Licensed by Texaco Development Corporation, 135 East 42nd Street, New York.

Desulfurization of Various Gasolines

	Desulf. of S. R. Gasoline from New Mexico Crude**		Desulf. of S. R. Gasoline from Michigan Crude**		Desulf. of Gasoline from Illinois Crude**		Desulf. of 50-50 Blend of Untreated Cracked Gasoline and Straight Run Gasoline from Michigan Crude***	
	H_2S Free Charge	H_2S Free Product	H_2S Free Charge	H_2S Free Product	H_2S Free Charge	H_2S Free Product	H_2S Free Charge	H_2S Free Product
Gravity, A.P.I.	55.5	55.6	67.8	67.6	.....	.....	64.8	64.2
Distillation:								
IBP	115	114	98	98	132	131	98	100
10 percent	169	173	135	134	188	187	136	140
50 percent	266	261	218	216	263	261	228	230
90 percent	387	397	290	286	349	348	320	330
EP	436	442	338	342	396	395	360	384
ASTM Gum	1	2	.....	.....	.....	.....	1.6	2.0
	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent
Total Sulfur	0.138	0.0280	0.053	0.005	0.038	0.006	0.105	0.042
Mercaptan Sulfur	0.057	0.0017	0.018	0.0004	0.007	0	0.040	0.000
Non-Mercaptan Sulfur (by diff.)	0.081	0.0263	0.035	0.0046	0.031	0.006	0.065	0.042
Doctor Test	Positive	Positive*	Positive	Negative	.....	.....	Positive	Negative
ASTM Octane No.:								
Clear	54.4	56.6	48.8	49.4	47.8	50.3	58.8	60.4
Plus 1cc TEL	61.3	67.2	59.2	62.6	58.1	62.4	65.1	67.0
Plus 2cc TEL	66.3	73.0	63.9	69.0	63.7	70.0	67.8	71.0
Plus 3cc TEL	69.4	77.2	67.0	73.1	68.0	74.0	70.3	74.3
1939 Research Octane No.								
Clear	56.5	59.6	.....	.....	.....	.....	.....	.....
Plus 1cc TEL	63.4	68.7	.....	.....	.....	.....	.....	.....
Plus 2cc TEL	68.8	73.4	.....	.....	.....	.....	.....	.....
Plus 3cc TEL	71.5	76.9	.....	.....	.....	.....	.....	.....
HR&G Lead Susceptibility	1.19	1.8	1.31	1.90	1.57	2.10	0.9	1.15
Clear Octane Increase:								
ASTM	.....	2.2	.....	0.6	.....	2.5	.....	1.6
1939 Res	.....	3.1	.....	.....	.....	.....	.....	.....
TEL Required for 63 O. N	.....	.....	2.35cc	1.35cc	.....	.....	.....	.....
TEL Required for 68 O. N	.....	.....	.....	.....	3.0	1.7	.....	.....
TEL Required for 69 O. N. ASTM	2.80cc	1.25cc	.....	.....	.....	.....	.....	.....
TEL Required for 70 O. N	.....	.....	.....	.....	.....	.....	3.0cc	1.6cc
TEL Required for 70 O. N. (Extr.)	.....	.....	4.20cc	2.20cc	.....	.....	.....	.....
TEL Required for 71 O. N. 1939 Res	2.70cc	1.40	.....	.....	.....	.....	.....	.....
TEL Required for 72 O. N. (Extr.)	.....	.....	.....	.....	.....	.....	4.0cc	2.2cc
TEL Saving at 63 O. N	.....	.....	.....	1.0	.....	.....	.....	.....
TEL Saving at 68 O. N	.....	.....	.....	.....	.....	1.3	.....	.....
TEL Saving at 69 O. N. ASTM	.....	1.55cc	.....	.....	.....	.....	.....	.....
TEL Saving at 70 O. N	.....	.....	.....	.....	.....	.....	.....	1.4cc
TEL Saving at 70 O. N. (Extr.)	.....	.....	.....	2.0	.....	.....	.....	.....
TEL Saving at 71 O. N. 1939 Res	.....	1.30cc	.....	.....	.....	.....	.....	.....
TEL Saving at 72 O. N. (Extr.)	.....	.....	.....	.....	.....	.....	.....	1.8cc

* Negative after Caustic Wash. ** Catalyst Life Approximately 5,000 barrels per ton. *** Catalyst Life 800 barrels per ton.


CLAY DESULFURIZATION UNIT
CLAY TOWER
H_2S GAS TO HEATER
EXCHANGER
HEATER
CONDENSER
COOLER
ACCUMULATOR DRUM
GASOLINE TO H_2S WASHER
CHARGE IN
CHARGE PUMP
TRANSFER PUMP
COURTESY GRAY PROCESSES CORPORATION

Gray Clay Treating Process

TEXACO DEVELOPMENT CORPORATION

THIS Process, named after its inventor, the late T. T. Gray, was first put into commercial operation in 1924 at Barnsdall, Oklahoma. The licensed Gray "clay treating" process is used in treating gasoline produced by various cracking, reforming, and polymerization processes. These Gray towers have treated the product of Cross, de Florez, Dubbs, Gyro, Holmes-Manley, Tank & Tube, Combination Units and others, from variegated charging stocks ranging from Pennsylvania gas oil to naphthenic base crudes.

The process is based on the selective polymerization of the unstable, unsaturated hydrocarbons resulting from pyrolysis. The gum forming and other objectionable properties of the gasoline are eliminated, while the desirable constituents pass through the process unchanged. The result is that treating losses are reduced to the vanishing point, with the octane number of the gasoline being processed unchanged. Most Gray installations are made in direct connection with the cracking unit whereby the rerunning step is eliminated and a treated gasoline is produced directly at the cracking plant. Vapors to be treated are passed through fuller's earth or similar adsorbent material, and the unstable unsaturated hydrocarbons are thereby polymerized into higher boiling compounds which are separated from the treated gasoline by virtue of their higher boiling point.

Treating costs are low. Operating costs are often only a fraction of a cent per barrel. No added labor is required and 3,000 to 30,000 barrels of gasoline have been treated per ton of clay before replacement or revivification is required.

The apparatus required is simple. Fuller's earth (usually 30-60 mesh) is charged to one or more of the treating towers (polymerizers) through which the vapors are passed. The greater part of the polymers formed separate from the vapors in the tower, and any remaining polymers may be removed by a dephlegmating or fractionating step before condensation of the treated vapors. Division of the fuller's earth into a series of beds gives better results than where the clay is used in one bed. However, except in cases where extremely low gum is required, a single treating tower is ordinarily sufficient. The flow diagram presented on the opposite page through the cooperation of The Gray Processes Corporation, Jersey City, New Jersey, shows a typical arrangement where two polymerizers are employed. The towers are so manifolded that either can be used first, or, if desired, they can be used in parallel.

The polymerizers are usually operated at the condensing pressure of the distillation unit to which they are connected. It has been found that the higher the pressure, the greater the throughput per ton of clay, all other things being equal. Commercial units have been successfully operated at pressures as high as 375 to 400 pounds.

The quality of gasoline made during a run is quite uniform, since the decline in activity of the clay is gradual. Often, the activity of the treating material can be effectively prolonged by passing steam or a gasoline spray through the treating towers for a short time. Plants operate 90 days or longer between changes of clay.

Following are results obtained in a typical installation:

RESULTS ON REFINING A HOLMES-MANLEY CRACKED GASOLINE FROM KENTUCKY CRUDE

Cracking Plant	Holmes-Manley Redistilled Circulation
Charging	Topped Kentucky Somerset Crude (Gravity 28.4° API)
Clean Oil Transfer Temp.	965-980° F.
Two Gray Towers in series	174 lbs. pressure top 1st Tower
Type of Clay	Olmstead
Throughput when sampled	3627 barrels per ton
Tests on Gasoline:	
Gravity	61.7°F. API Color-Saybolt 30+
Distillation: IBP	84°F. Octane 78-77
10%	124°F.
20%	...
50%	230°F.
90%	357°F.
End-point	396°F.

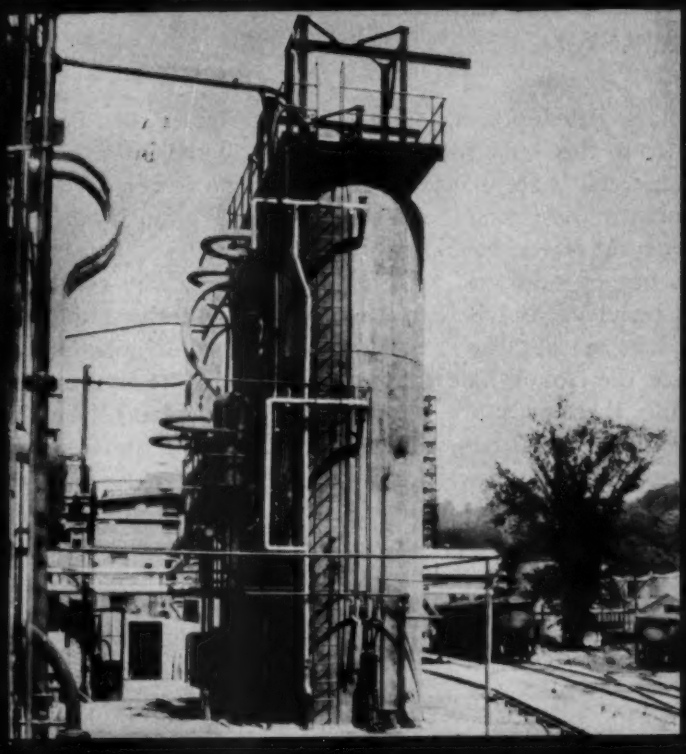
Gum Tests:	Doctor Sweetened
Copper Dish (mg./100 ml.)	3
Oxygen Bomb: Induction Period (hrs.)	2½

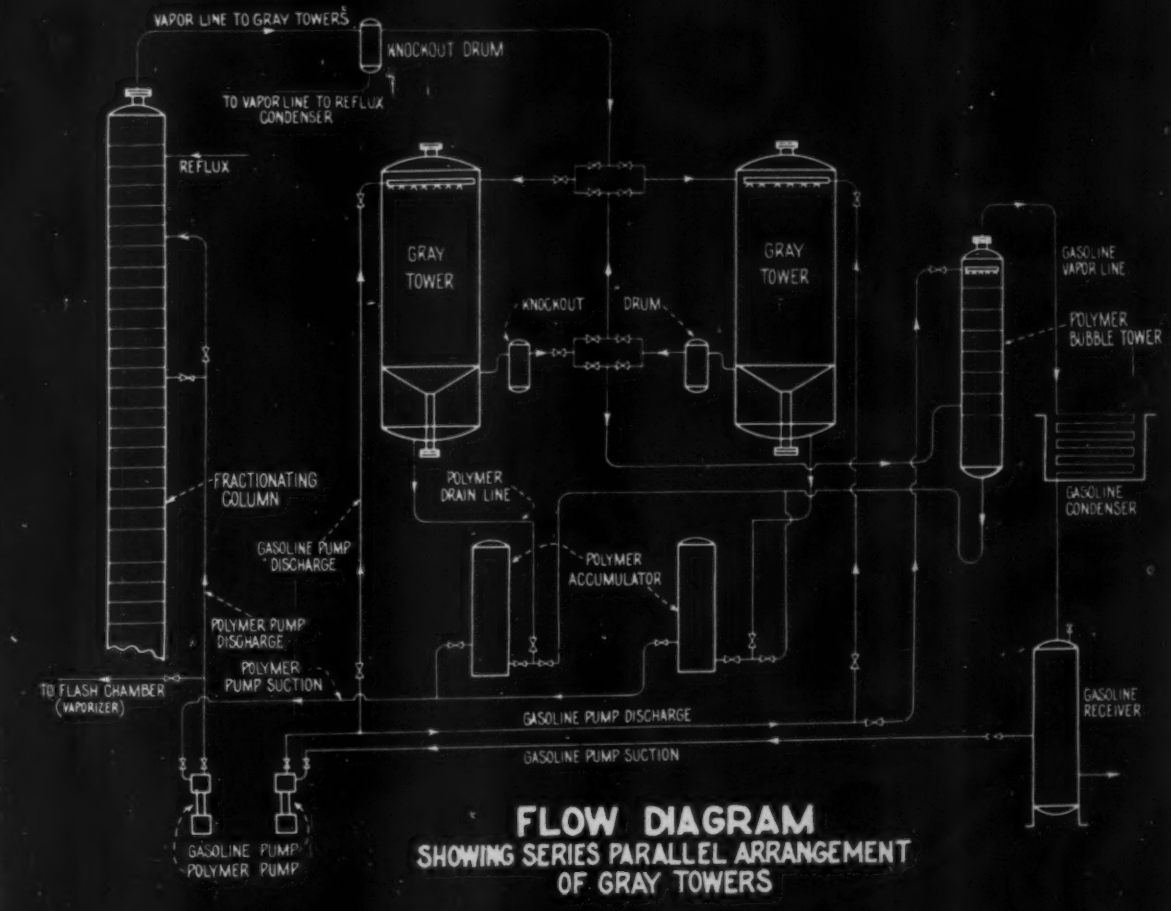
Storage Behavior of Gasoline (Doctor Sweetened Samples):

Months Stored	1	2	3
Copper Dish Gum (mg./100 ml.)	2	2	4
Color Saybolt	+30	+29	+29

Note the low copper dish gum test and good storage behavior.

The Gray Process is licensed by Texaco Development Corporation, 135 East 42nd Street, New York.





FLOW DIAGRAM
SHOWING SERIES PARALLEL ARRANGEMENT OF GRAY TOWERS

COURTESY, THE GRAY PROCESSES CORPORATION

Hot-Acid Polymerization

SHELL DEVELOPMENT COMPANY

THE Shell Companies have developed a method for producing isooctane from the butylenes present in refinery gas. Commercial size plants have given satisfactory performance over many months and the results in different refineries have proved the merit of this catalytic use of sulphuric acid. In this process both normal butylene and isobutylene hydrocarbons react to produce isooctenes. The yield of octenes obtained is twice the isobutylene content of the feed stock providing there is more normal butylene present in the original stock than isobutylene, as is usually the case. When these octenes are hydrogenated the resultant product has an octane number closely approaching that of pure isooctane. Feed stocks in different refineries are seldom the same, but the process has operated successfully on C_4 fractions varying widely in content of normal and isobutylene.

The flow diagram on the opposite page shows the major pieces of equipment involved in the hot-acid process. The charge of butane-butylene fraction enters the system continuously through the recycling line of hydrocarbon and acid. Fresh acid is also added to this recycling line. The mixture of recycled stock, fresh acid and butane-butylene fraction goes first to a reactor cooler and then to a reactor time tank where the polymer is produced. The reaction is exothermic but sufficient cooling is provided to maintain a temperature of about 80° C. This temperature promotes rapid polymerization so that the absorbed butylenes have only a transitory life in the acid phase, the polymer being immediately reabsorbed in the hydrocarbon phase. The process is thus truly catalytic. Present practice is to use about 70% acid and to allow from 10 to 15 minutes time of contact.

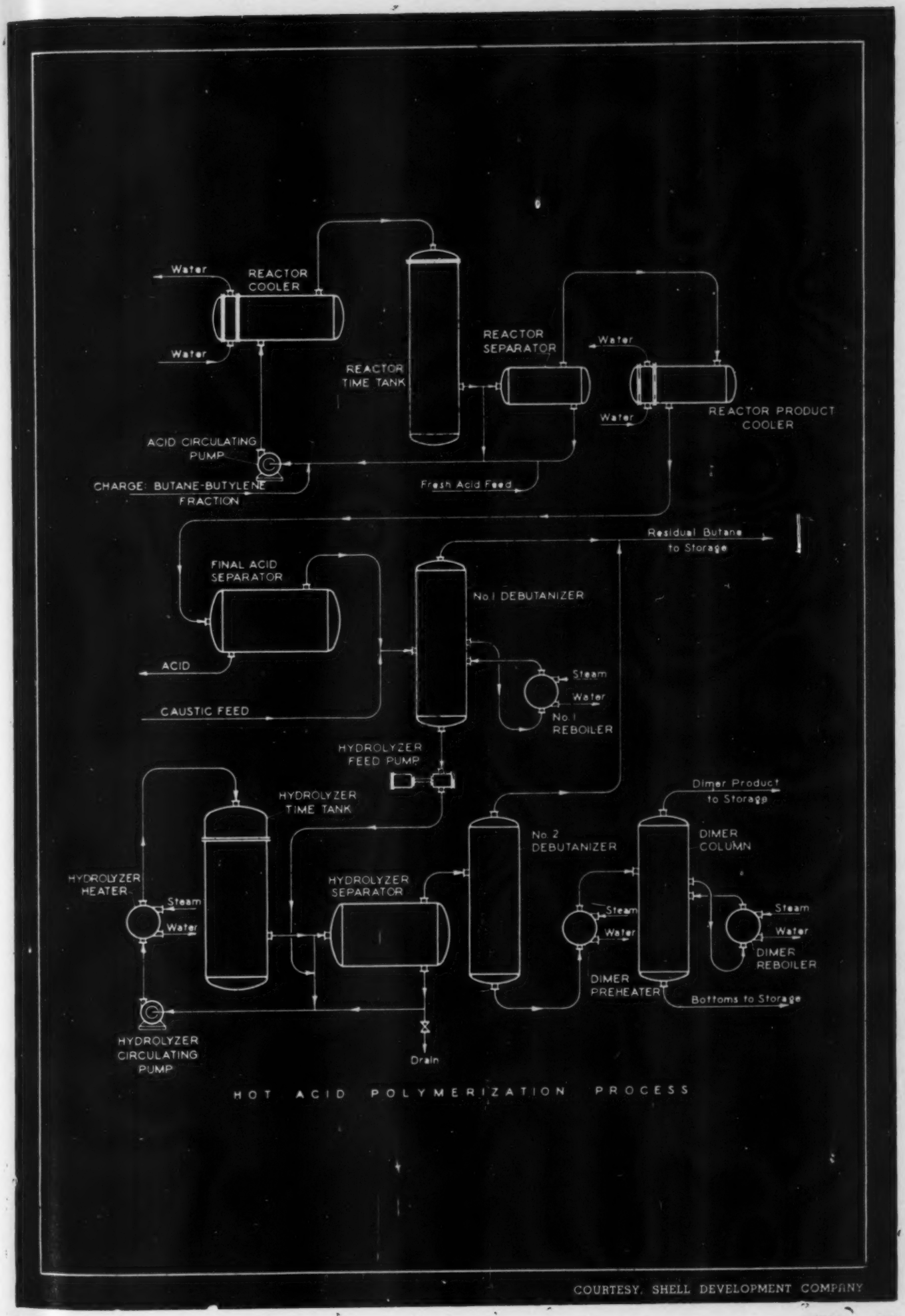
The mixture of hydrocarbon, polymer and acid leaving the reactor time tank flows into the recycle line and into a reactor separator where most of the acid is removed by gravity separation. The stream of hydrocarbon and polymer from the separator is further cooled in the reactor product cooler and passed into a final acid separator. The hydrocarbon and polymer then flow to the first debutanizer where most of the butane and some of the surplus normal butylene is removed overhead.

The bottom product containing all the polymer is then delivered to a recycle line from the hydrolyzer time tank. The recycle line flows to a heater using exhaust steam and thence to hydrolyzer time tank in which all entrained acid is neutralized and acid sulphates are hydrolyzed with caustic. The caustic-polymer mixture then passes to a hydrolyzer separator where the free caustic is removed, the hydrocarbon and polymer residue being delivered without further heating to a second debutanizer. In this column the remainder of the butane and all surplus butylene is removed, the bottoms being pumped through another steam heater to the dimer column. Here the dimer, which is the final product, is separated from the heavier hydrocarbon and delivered overhead to storage.

The chief advantage of this system is that normal butylene as well as isobutylene is used to produce the polymer (or dimer). Normal butylenes do not polymerize with themselves as readily as do isobutylenes, but in the presence of isobutylene the normal butylenes will react readily to form interpolymer and it is for this reason that the yield of polymer is equivalent to twice the amount of isobutylene present in the charging stock. The interpolymer, passing into the hydrocarbon phase as rapidly as it is formed, is diluted by the inert butanes. Its activity is thus reduced, leaving little opportunity for the formation of trimers or rearrangement to polymers of lower octane rating. Consequently, the polymer formed is about 90 to 95 percent octenes. The product obtained upon hydrogenation has an octane number averaging between 98 and 99.

Operation of the unit is simple; one operator per shift is sufficient. Both polymerization and neutralization are automatic once the rate of flow is established.

This process is licensed by Shell Development Company, New York.


Water
REACTOR COOLER
Water
REACTOR TIME TANK
REACTOR SEPARATOR
Water
Water
REACTOR PRODUCT COOLER
ACID CIRCULATING PUMP
CHARGE: BUTANE-BUTYLENE FRACTION
Fresh Acid Feed
Residual Butane to Storage
FINAL ACID SEPARATOR
No. 1 DEBUTANIZER
ACID
Steam
Water
No. 1 REBOILER
CAUSTIC FEED
HYDROLYZER FEED PUMP
Dimer Product to Storage
HYDROLYZER TIME TANK
No. 2 DEBUTANIZER
DIMER COLUMN
HYDROLYZER HEATER
Steam
Water
HYDROLYZER SEPARATOR
Steam
Water
Steam
Water
DIMER REBOILER
DIMER PREHEATER
Bottoms to Storage
HYDROLYZER CIRCULATING PUMP
Drain
HOT ACID POLYMERIZATION PROCESS
COURTESY, SHELL DEVELOPMENT COMPANY

Houdry Dehydrogenation Process

HOUDRY PROCESS CORPORATION

THE Houdry dehydrogenation process employs the "adiabatic" principle, eliminating heat exchange within the reactor and resulting in considerable simplification of equipment. At the same time adequate temperature control is provided to insure high conversion and good selectivities. It is equally applicable to the production of mono-olefins and di-olefins from their corresponding paraffins, di-olefins from mono-olefins or from mixtures of paraffins and mono-olefins and aromatics with unsaturated side chain from alkylaromatics.

Dehydrogenation is accomplished by passing the preheated hydrocarbon vapors under controlled conditions of space velocity, temperature, and pressure over catalysts of suitable composition and controlled activity. During the reaction period, a large amount of heat is absorbed due to the endothermic heat of reaction. In addition to products of dehydrogenation, including free hydrogen, a small amount of fixed gas is produced and coke is deposited on the catalyst. This coke has to be burned periodically in order to restore initial catalyst activity.

The characteristic feature of the Houdry dehydrogenation process is that the operating conditions are so chosen that the heat used for the reaction is substantially balanced by the heat developed from the combustion of the coke deposit. Furthermore the heat capacity of the catalyst bed is controlled by mixing with the active catalyst an inert material of granular form having high density and high specific heat. This inert material acts as a heat-storage medium, absorbing, during the regeneration cycle, the heat of combustion of the coke deposit with a resultant increase in temperature and releasing this heat during the reaction period with corresponding temperature decrease. A combination of short on-stream periods and high heat capacity of the catalyst mass insures a narrow range of temperature necessary for optimum conversion and selectivity, while adjustments in the desired temperature levels are obtained by variation of hydrocarbon and air inlet temperatures.

Advantages derived from the use of this scheme are as follows:

(1) Cheap reactor construction since no special-alloy heat-exchange equipment is necessary. Carbon steel, internally insulated, can be used.

(2) Reactants are introduced at substantially reaction temperature, precluding thermal cracking.

(3) No high temperatures are encountered during regeneration, thus catalyst life is extended.

(4) Regeneration can be accomplished with fresh air. No flue-gas recirculation is necessary.

(5) Due to adequate temperature control, optimum conversion and selectivities are maintained.

Mechanically the process operates under the principles of the Houdry Fixed-Bed Cracking Process, with reactors arranged in groups of three. On each reactor, the on-stream period is followed by a vacuum purge of hydrocarbons then a regeneration period accomplished by circulation of air preheated by direct combustion of fuel in the stream. At the end of the regeneration period products of combustion also are purged by means of a steam ejector, prior to the following on-stream period. Thus in a three-reactor group one reactor receives hydrocarbons while the second receives air and the third undergoes valve changes and purging operations. By these means, continuous flow of air and hydrocarbons is achieved.

Motor valves are operated by a cycle timer of conventional design and electrical interlocks preclude operation of a particular valve if the operation of the preceding valve has not been completed.

The products of reaction are cooled, separated from the fixed gases and purified in equipment of conventional design, unconverted material being returned as cycle stock to the process.

Several years of continuous operation of two units producing butadiene from normal butane have demonstrated the commercial feasibility of the process.

HOUDRY DEHYDROGENATION PROCESS
SIMPLIFIED FLOW DIAGRAM
COURTESY, HOUDRY PROCESS CORPORATION
F. FEED
HEATER
STEAM JET
AIR OUT
AIR HEATER
AIR BLOWER
REACTORS
QUENCH TOWER
RECYCLE STOCK
EXTRACTION AND PURIFICATION
STEAM
FINISHED PRODUCT
STEAM
FUEL GAS
RECOVERY
COMPRESSION

Houdry Fixed-Bed Catalytic Cracking Process

HOUDRY PROCESS CORPORATION

IN THE field of catalytic cracking the Houdry process employing fixed catalyst beds is the pioneer. The first commercial-scale plant was put in operation in 1936 and since then many additional units have been installed in both the United States and abroad.

Two fundamental needs of catalytic cracking are to furnish heat during the endothermic cracking period and to remove heat during the exothermic regeneration, or the period during which the carbonaceous catalyst deposit formed from the cracking reaction is burned. The fixed-bed type reactor or case has been designed to fulfill these requirements.

Essentially, the reactors are tubular heat-exchange vessels. The catalyst bed lies in the shell side through which flow the oil or air vapors. On the tube side a molten mixture of alkali metal salts is circulated. Inasmuch as the net effect of catalytic cracking and carbon burn-off is exothermic, the circulated salt is used as a cooling medium. The high heat capacities of the steel reactor structure and of the catalyst bed have been major factors in design of equipment.

Full utilization is made of the large quantities of heat evolved during operation. The bulk of the heat is removed from the reactors by the flowing molten salt, of which a part is circulated through a by-pass to the salt-steam generator and superheater and another portion through a salt-air heat exchanger used for preheating the regeneration air to the cases. The relatively cool salt returning from the boiler and air heater is fed again into the main body of salt, and by this means the total salt to the reactors is maintained constant at any desired temperature. The heat carried from the reactors by the regeneration gases is utilized in providing power to the gas turbine used to drive the compressor supplying regeneration air.

A major improvement in the Houdry fixed-bed process has been affected in the past two years by redesigning the reactor tubes and changing the physical composition of the catalyst mass. The heat-exchange conditions have been improved and mechanical and process advantages obtained. Since the new design is a simplification of the original, lower operating costs result by virtue of reduced maintenance and downtime for catalyst change and turnarounds. All vapor distributing elements are now easily removable from the cases and are, therefore, removed and thoroughly cleaned on every catalyst change, assuring good distribution of the vapors and maximum yields of the desired cracked products. Because of the improved mechanical distribution thus attained and the higher on-stream efficiencies, greater production of high-octane gasoline results.

Three 12-case Houdry units have been converted to the new design and one has been in operation for two years. It is reported that not only are operating costs reduced and gasoline output increased, but that improved gasoline quality has been achieved because of the greater supply of heat available within the catalyst mixture for the cracking reaction.

The flow diagram shows the three essential parts of the Houdry system—(1) a furnace for vaporization of the charge to the reactors, (2) the catalytic or case section, and (3) the fractionating equipment for splitting the synthetic crude into useful products. Only three catalyst cases are shown, illustrating the least number generally employed in Houdry cracking plants. Larger plants employ multiples of three or four cases with the reactors manifolded in pairs.

Either natural or synthetic catalyst may be loaded to the reactors and in those plants modified to the new design mixtures of active catalyst and high-heat-capacity, low-cost inert material have been used. In the modified reactors essentially the same quantity of active catalyst is charged as before design changes were made. The inert material takes the place of those tubes and metal fins that were eliminated in the simplified design.

An electrical timer transposes the cyclic operation of each reactor to a continuous operation of the overall unit. The cycle is, briefly, that one reactor or pair is on-stream while a second is being regenerated and the third is on oil purge, air purge, or repressuring operation and undergoing the necessary valve changes. The purges are affected by steam-jet ejectors and, in addition, during the oil evacuation, sweeping steam is admitted to the reactor. Motor-operated valves actuated by impulses from the cycle timer assure continuous oil and air flow through the system, and appropriate electrical interlocks preclude any mixing of the streams.

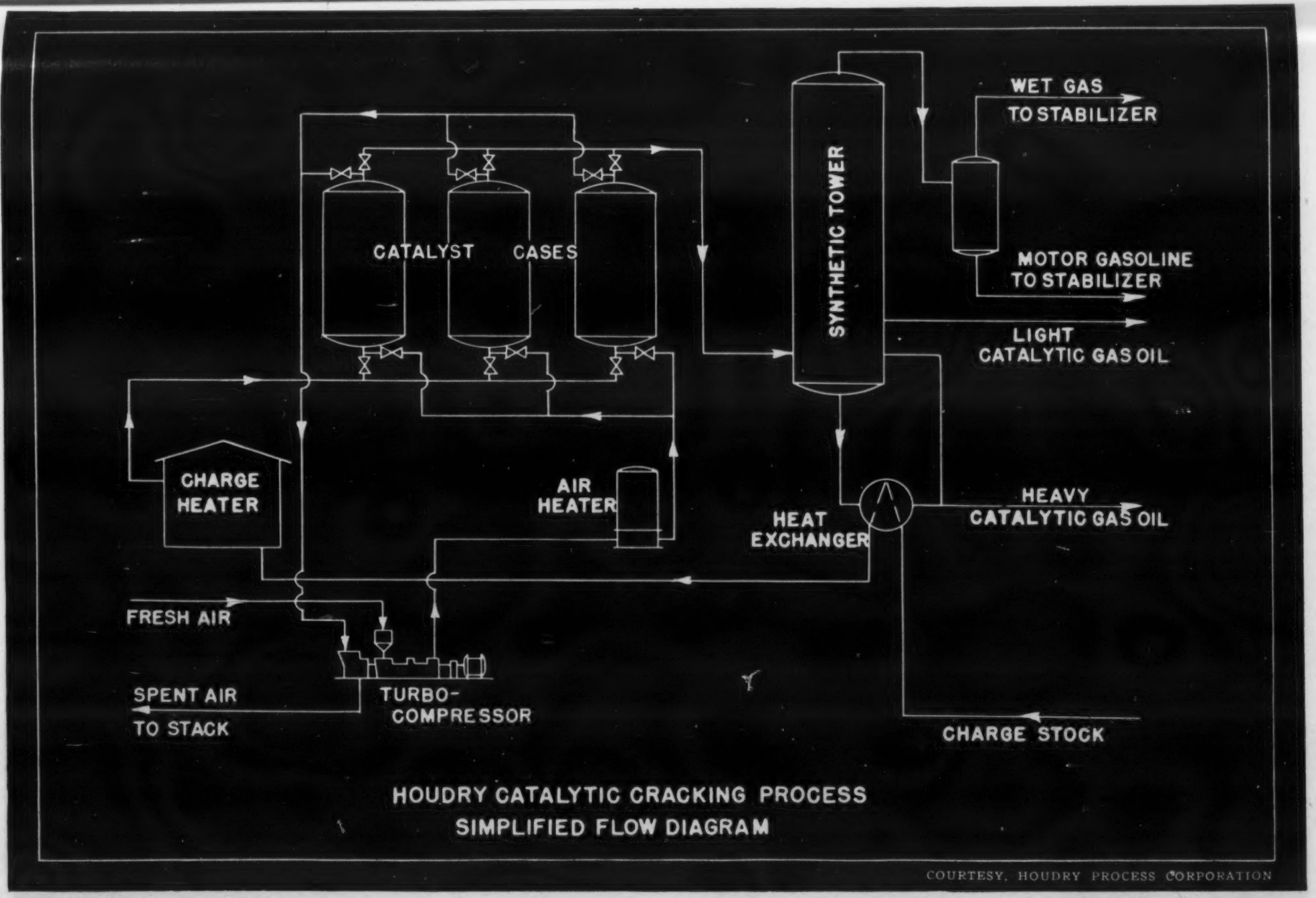
By adjustment of the case operating variables such as temperature, pressure, space rate, etc., the relative yields of gasoline, fuel gas and catalytic gasoil may be varied over a wide range. Typical operating conditions, yields, and inspections when cracking gasoil for motor gasoline production are given in the accompanying table.

Typical Houdry Fixed-Bed Operation Cracking Gasoil from Mixed Base Type Crude

Type Process—Once-Through Cracking for Motor Gasoline Production
Type Charge Stock—Gasoil from Mixed-Base Type Crude
Type Catalyst—Synthetic

Operating Conditions:	
Space Velocity, V/Hr/V	1.0
Temperature of Salt to Reactors, °F.	810
On-Stream Reactor Pressure, psig	15
Process Steam	None
Time on Stream, Minutes	10
Time of Complete Cycle, Minutes	30

	Vol. Percent Fresh Charge to Reactor	Gravity °API	5 Percent	10 Percent	50 Percent	90 Percent	95 Percent	R.V.P. (psi)	C.F.R.M. Clear Octane	Saybolt Color
Charge:										
Virgin Gasoil	100.0	35.2	490	506	560	672	710	....	....	....
Products:										
Motor Gasoline	46.3	57.9	115	127	232	368	....	9.9	78.9	30
Catalytic No. 2 Fuel Oil	39.8	32.5	470	480	520	590	610	....	....	....
Catalytic Heavy Gasoil	5.0	18.0	588	600	650	710	....	....	....	....
Excess C_4 Fraction	9.9	.....	....	....	.....	....	....	....	....	....
Dry Gas, C_3 & Lighter, Weight Percent	4.8	....	....	....	....	....	....	....	....	....



HOUDRY CATALYTIC CRACKING PROCESS
SIMPLIFIED FLOW DIAGRAM

COURTESY, HOUDRY PROCESS CORPORATION

Houdry Process for Treating Motor Gasoline

HOUDRY PROCESS CORPORATION

THE Houdry Process for treating motor gasoline is a catalytic, vapor-phase, low-temperature, regenerative process for handling straight-run, thermally-cracked or reformed, and catalytically-cracked motor gasolines, or blends of such gasolines. It is applicable to low-sulfur and high-sulfur-content gasolines, yielding a product which, after fractionation to end-point and stabilization, is sweet to doctor test, non-corrosive to copper strip, of good color and color stability, of low gum content, and stable to oxidation. It also results in improved leaded octane ratings and does not depreciate but in many instances improves unleaded ratings.

Although there is always a reduction of the total sulfur content, the process is especially selective to sulfur compounds which react to the doctor and copper-strip tests, and effects practically complete removal of these compounds from high- and low-sulfur gasolines. The less active sulfur compounds remain in the gasoline, while the sulfur compounds removed are retained on the catalyst and rejected along with the products of combustion during the regeneration cycle.

Colored, color-forming, gum and gum-forming components are converted to polymers, the heaviest of which are deposited on the catalyst while the balance is carried from the catalyst with the other products of reaction and separated by fractionation to be recovered as a polymer or light gasoil fraction.

Yields of treated gasoline are in the range of 95-98 percent by volume, depending on the quality of the charging stock. Polymer gasoil, catalyst deposit and a small quantity of by-product gas comprise the balance of the products.

Typical results from application of the process to high- and low- sulfur gasolines are given in the accompanying table which presents data obtained in laboratory pilot-plant and current commercial operation.

The process is effected in salt-cooled fixed-bed reactors. The operating cycle comprises the on-stream period, the duration of which depends on the charging stock but is always several hours; a purge period effected by steam circulation; a regeneration period during which low-pressure preheated air is introduced to the reactor to burn off the coke deposit left on the catalyst by the preceding on-stream period, and molten salt is circulated to remove the heat of combustion and bring the reactor back to operating temperature; and a final steam-purge period, prior to the introduction of hydrocarbons. Due to the length of the on-stream period, only two reactors are necessary: the flow of oil and air are continuous, but the air is by-passed during the purges and valve changes.

The raw, unstabilized charging stock is brought to operating temperature, of the order of 600-650° F. in a conventional furnace after heat exchange with products of reaction. Operating temperature is maintained during the on-stream cycle by the heat of the stream and a slightly exothermic heat of reaction. The products are partially cooled in exchangers and are then conveyed to a fractionator where polymer is separated and final adjustment in end-point is made. The overhead then is stabilized to the desired vapor pressure.

During the regeneration cycle the catalyst reaches a maximum of 1000-1100° which is controlled by the rate of air flow and the circulation of molten salt. At the end of the regeneration period salt circulation is continued to bring the reactor to operating temperature. The heat of combustion is recovered as high-pressure steam in a boiler of special design, wherein heat is removed from the circulating molten salt.

Reactor design is adapted to these conditions and is somewhat different from the conventional cracking reactors, allowing regeneration temperatures suitable for catalyst clean-up, and lower on-stream temperatures. The catalyst is especially prepared to fulfill the dual purpose of sulfur retention and polymerization of undesirable components. Commercial experience has indicated a catalyst life of several years.

The operation of two large commercial units has been completely satisfactory for several years on blends of catalytically-cracked, thermally-cracked and thermally-reformed gasolines.

Houdry Treating High- and Low-Sulfur Gasolines

CHARGE STOCK	Thermally Cracked Gasoline		Thermally Cracked Gasoline		Blend of Thermal and Catalytic Gasoline		Blend of Thermal and Catalytic Gasoline	
Type Operation	Pilot Plant		Pilot Plant		Pilot Plant		Commercial	
Yields:								
Treated Gasoline, Percent Vol.	96.3		96.9		95.9		98	
Polymer, Percent Vol.	1.9		1.00		2.5		1.6	
Gas, Percent Weight	.53		.47		.46		.2	
Coke, Percent Weight	1.25		1.48		.62		.55	
Inspections:	Charge	Product	Charge	Product	Charge	Product	Charge	Product
Distillation, ASTM:								
I.B.P.	109	114	98	110	101	120		
10 Percent	157	165	168	164	160	172		
50 Percent	266	266	268	278	288	284		
90 Percent	370	364	387	378	378	374		
E.P.	420	409	416	420	415	414		
Recovery, Percent	98	98	97	97	98	98		
Color, Saybolt	—13	25	6	27	(Yellow)	30	0—	27-28
Doctor Test	sour	sweet	sour	sweet	sour	sweet	sour	sweet
Copper Strip	tarn.	o.k.	tarn.	o.k.	o.k.	o.k.	tarn.	o.k.
Glass Dish Gum mg./100 cc.	—	—1	11	1	7	1	2-7	1
Copper Dish. Gum mg./100 cc	24	8	152	5	71	10	35-75	10-11
OxBomb Stability, Hrs.	...	...	...	10	...	10+		
Sulfur, Percent Weight	.77	.53	.36	.29	.05	.03		
Octane Rating F2:								
Clear	70.9	71.9	....	71.2	74.9	76		
1cc TEL/Gal.	74.7	75.4	....	74.5				
3cc TEL/Gal.	77.1	78.9	....	78.4				

FIXED GAS
GASOLINE TO STABILIZER
POLYMER
FRACTIONATOR
AIR OUT
OIL OUT
SALT OUT
SALT IN
REACTORS
STEAM
SALT BOILER
OIL IN
AIR IN
RAW FEED
HEATER
BLOWER
AIR HEATER
HOUDRY PROCESS FOR TREATING MOTOR GASOLINE
SIMPLIFIED FLOW DIAGRAM
COURTESY, HOUDRY PROCESS CORPORATION

Howard Treating Process

PETROLEUM RESEARCH CORPORATION

THE Howard Process is used in the treatment of light petroleum distillates—both cracked and straight-run products. It is licensed by Petroleum Research Corporation, 916 Walnut Street, Kansas City, Missouri, with research and development laboratory work.

In its essence, it is a vapor-phase treating process, comprising a single once-through operation which depends upon a consecutive series of chemical and catalytic reactions and interactions to efficiently perform the treating functions. The mechanical equipment is so arranged as to permit the various reactions to proceed under conditions favorable for effecting those results desired. The following functions are effected, namely: removal of objectional gums and gum forming materials; excellent stabilization toward oxidation, and sunlight; production of negative doctor and negative corrosion; appreciable sulphur reduction; production of good color and color stability; retention of inherent octane value; appreciable improvement in tetraethyl lead susceptibility; and, low treating losses.

Briefly, the process comprises a system wherein the raw vapors are first subjected to intimate and uniform treatment with chlorine gas; providing for removal of any resultant polymerized fractions from the vapors; then leading the treated vapors into a contact vessel where they intimately contact a solid reagent, the active ingredient of which is an oxidized form of zinc, metallic zinc, or a mixture of both; and finally condensing the vapors. The active ingredient in the contact vessel is carried in a solid binder, in which it is uniformly dispersed. The contact material is a manufactured product of specification materials. All heavy end fractions and polymers return to a prior point in the plant system where they are reflashed to recover their gasoline content, and the heavier fractions pass, respectively, into the gas oil and fuel oil. The condensed gasoline is led to storage.

Operating temperatures are those inherent to the vapors at system pressures ranging between 15 and 25 lbs. gage. Automatic controls can readily be applied where required. Treating is effected with a minimum of chemical reagent, chlorine consumption varying between 0.05 lb./bbl. and 0.25 lb./bbl., while the contact bed can be periodically reactivated by simple steaming, and, when required, by burning in place.

A number of applications of the process are permissible and any specific installation is governed principally by the type of stock to be processed. Cracked distillates of a very refractory nature and carrying comparatively high sulphur contents are preferably stabilized first to eliminate propane, hydrogen sulphide, and excess butane, before being subjected to treatment. Milder types of cracked distillates lend themselves to processing in direct connected units, where the processing unit becomes an integral part of the cracking system. Insertion of the treating unit between the fractionating column and the condenser, providing for direct-connected operation, is prescribed in the treatment of straight run gasoline. The combined gasoline and kerosene vapor cut may be treated as a whole, with fractionation and separation of the gasoline and kerosene provided for in a following tower. Both the cuts will show major reduction of sulphur.

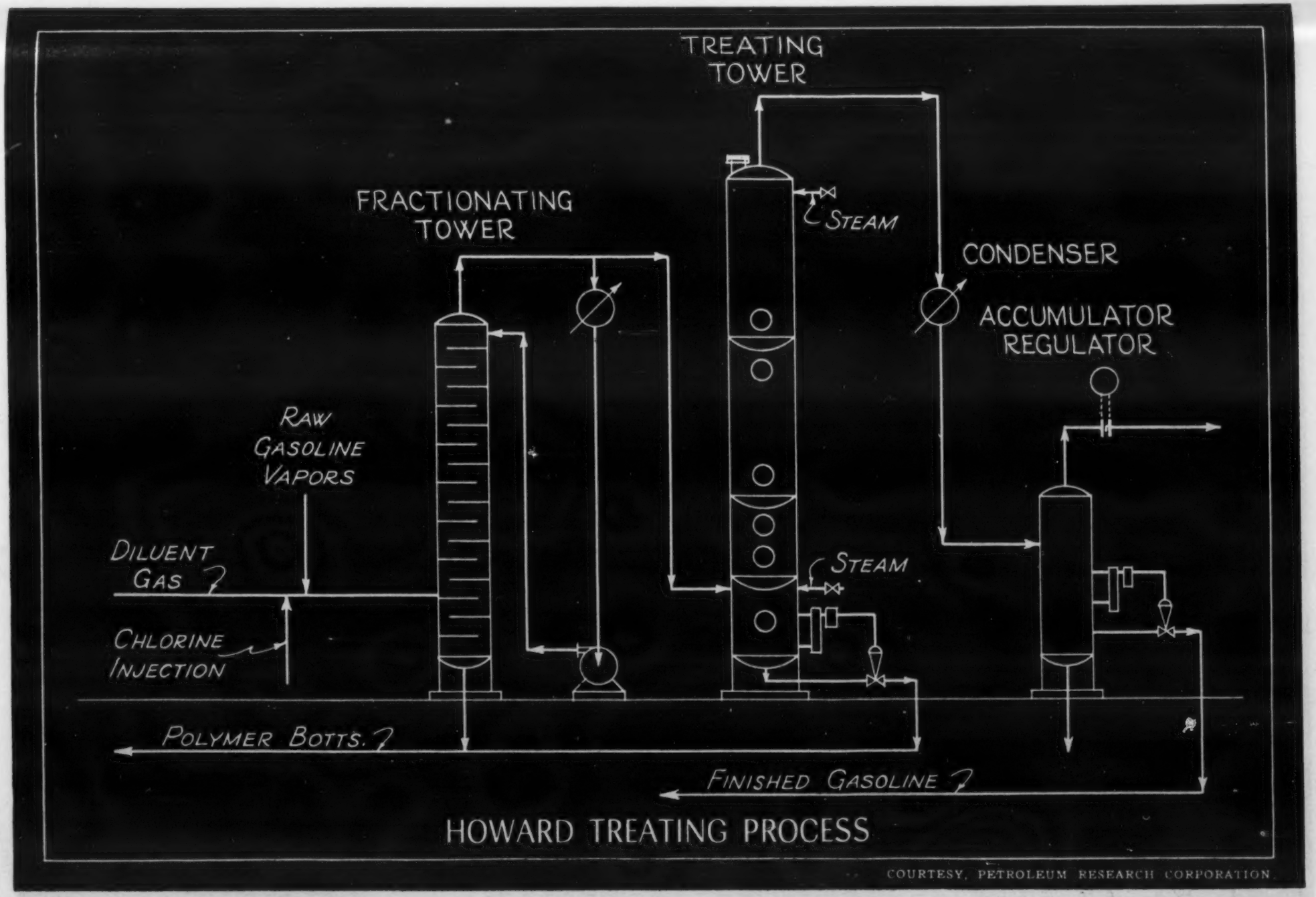
Many different types of products have been successfully treated. These products include cracked products produced from the conventional type cracking processes including vapor phase and polymerization units, and from stocks originating from a wide range of territories. Many straight run products have been treated, these ranging from comparatively low to comparatively high sulphur and mercaptan contents. In the once-through operation the process gives the combined results of a gum treatment, a sweetening treatment, sulphur reduction, color and gum stabilization, and improved lead susceptibility.

In the matter of gum removal, gasolines after treatment show contents of from 2 to 12 mgs. by copper dish, but seldom show any gum by glass dish. Different type stocks account for difference in gum content. Good stability is secured. The induction period has also been found to vary, according to the type of material treated. Vapor-phase gasolines and those produced by high temperature cracking show from 6 to 10 hours induction period, but the average cracked gasolines from the conventional types of cracking processes show generally higher induction periods, in some instances going to as high as 30 hours. The long induction period, being indicative of good storage stability, is of considerable interest to those storing finished gasoline for a considerable period.

Products recovered from the treating unit are 30 color, and it has been demonstrated many times that both cracked and straight run products are exceptionally stable to sunlight. No haze, cloud, or deposition upon the walls of the vessel occurs after many hours exposure. Effective elimination of deleterious sulphur compounds is obtained.

The sulphur reduction is appreciable, averaging on all products in the order of 50 percent. The sulphur reduction varies, however, with different stocks and the general average reduction is 10 percent more on straight run products than on cracked products. A minimum reduction of 15 percent has been found on some products while a maximum reduction of 80 percent has been found on others. The difference of chemical nature of the sulphur compounds in the various stocks account for the difference in sulphur reduction; some sulphur compounds being readily susceptible to this type treating. Numerous determinations have shown that the total sulphur reduction is always equal to and usually greater than the mercaptan sulphur content of the raw stock.

No degradation of the product either as to octane or lead susceptibility is occasioned by treatment. On the contrary, there is a marked improvement in most cases and the magnitude of the improvement is tied in with the sulphur reduction, and obviously closely related to it. Appreciable economies are therefore possible in those instances where additions of tetraethyl lead are used to improve octane. Savings on the order of 50 percent are shown on straight run gasolines, while cracked products show a variation of from as low as 10 percent to as high as 40 percent.



HOWARD TREATING PROCESS

COURTESY, PETROLEUM RESEARCH CORPORATION

Hydrocol Process

TEXACO DEVELOPMENT CORPORATION

THE Hydrocol Process, developed by Hydrocarbon Research, Inc., for the production of premium quality gasoline, diesel fuel oil and other products by the reaction of carbon monoxide and hydrogen, is a method for the synthesis of petroleum products. The starting material may be oxygen from the air and almost any hydrocarbon material, such as natural gas, coal, heavy crude oil or fuel oil. Due to its ability to use sources of carbon other than from petroleum to manufacture products such as motor fuel, the Hydrocol Process may be looked on as a means of greatly extending crude oil resources. Development of the process using natural gas as the raw material has brought it in line competitively with conventional refining processes, such as catalytic cracking, for the production of high-grade motor fuels. A number of large commercial installations are now under way.

For a number of years, particularly during the past war period, the Germans prepared a synthetic gasoline from carbon monoxide and hydrogen by the Fischer-Tropsch process. In this operation the Germans reacted steam with coke and then adjusted the ratio of hydrogen to CO to that desired in the synthesis step by passing the gas through a water-gas shift reactor which converted some of the carbon monoxide to carbon dioxide and hydrogen. The resulting gas freed from its CO_2 was then passed over a cobalt catalyst in a fixed bed. This process had many shortcomings:

1. The catalyst was expensive and reworking was frequent.
2. The catalyst was extremely heat sensitive; this coupled with the fact that enormous quantities of heat are liberated in the reaction made for complicated and expensive reactor design.
3. Power recovery from the reaction was low.
4. The gasoline produced was of poor quality (octane number 40 ASTM).

These drawbacks were so serious that the Fischer-Tropsch process could be justified economically only under an artificial economy. It appeared, however, that the basic reaction of CO and hydrogen to produce gasoline and other hydrocarbon products had a definite place in American economy. With that in mind, chemists and engineers of Hydrocarbon Research, Inc., and associates have been studying the process for more than eight years. The Hydrocol Process, as offered for license today, represents the culmination of these years of work. Its achievement depended upon the successful solution of the following problems:

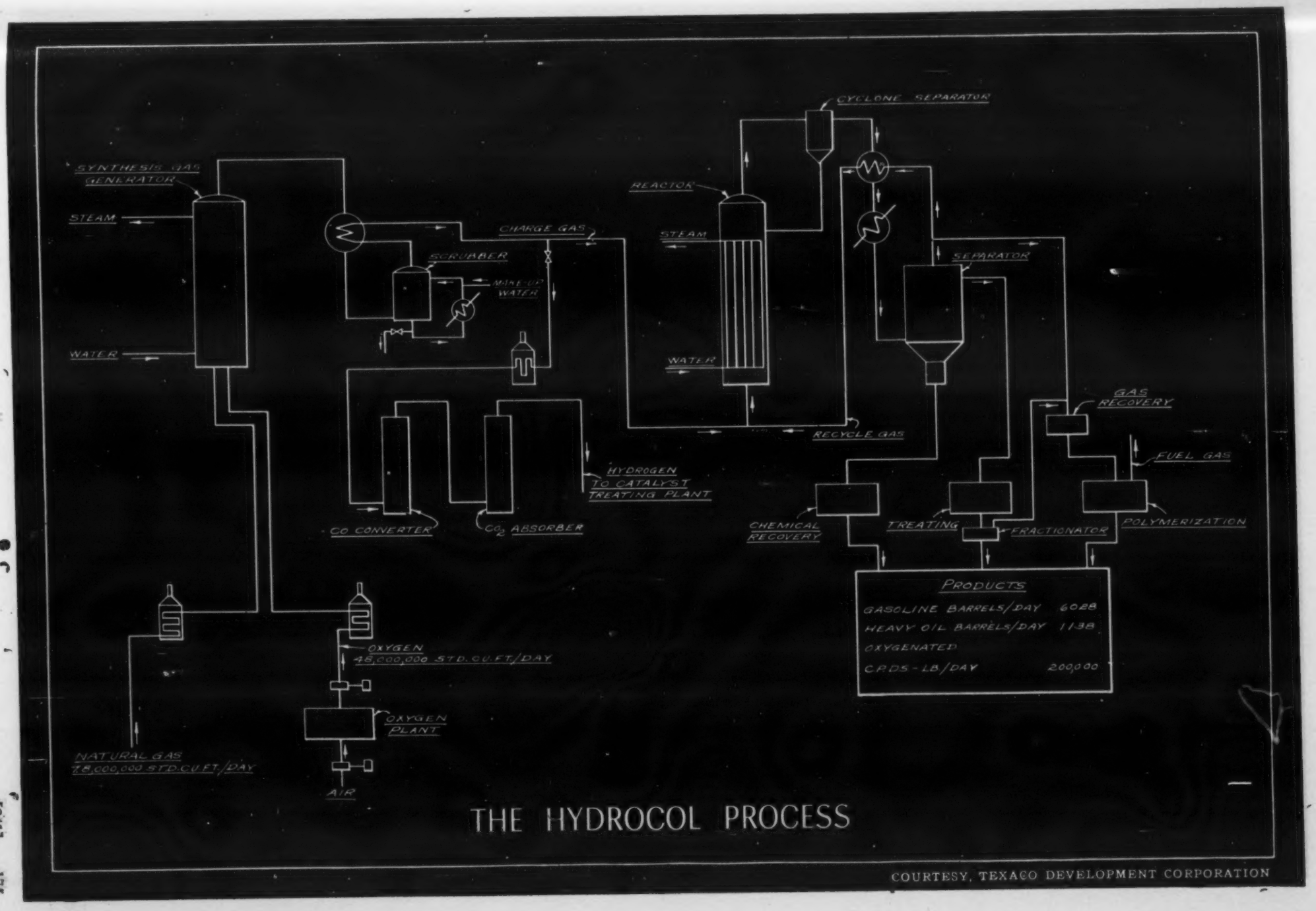
1. The manufacture of cheap oxygen.
2. The efficient production of Hydrocol or synthesis gas from natural gas and oxygen.
3. The development of a cheap, rugged and efficient catalyst to promote the conversion of Hydrocol gas into gasoline.
4. The engineering of an integrated plant, including a reactor design, which would permit absolute temperature control and an efficient recovery of the heat of reaction in the form of useful power.

A simplified flow diagram of the process as applied to one of the plants now under construction is shown on the opposite page. Natural gas and oxygen separately preheated are caused to react in an open-type generator at 2500° F. and 250 psig. The resulting gas is scrubbed with water and then heated by exchange with the generator gas and divided into a major and minor stream. The minor stream is passed over a water-gas shift catalyst to convert the CO to hydrogen and CO_2. The CO_2 is scrubbed out and the hydrogen is passed to the catalyst treating plant. The major stream is introduced into the synthesis reactor where it is caused to react in the presence of a stream of recycle gas at 650° F. and 245 psig over a fluidized bed of catalyst. The products leaving the reactor are passed to a cyclone separator from which catalyst fines are recovered. These products pass through a heat exchanger where they are employed to heat recycle gas. From the heat exchanger the products pass to a cooler and into a separator. In the separator, separation into three phases occurs, namely gas, hydrocarbon and water. A portion of this gas is passed to an absorber, while the remainder is recycled back to the reactor. The water containing by-product oxygenated compounds is drawn off the bottom of the separator and treated for the removal of the chemicals, consisting chiefly of alcohols, ketones, acids and aldehydes. The small percentage of by-product oxygenated compounds contained in the hydrocarbon fraction is removed and the product is then fractionated. The off gas is passed to an absorber with gas from the separator, and the total C_3s and C_4s are polymerized and the polymer gasoline is combined with the treated gasoline. The unabsorbed gas is used as fuel for the oxygen and natural-gas preheaters. In the synthesis step 98.8 percent of the CO and 95.1 percent of the hydrogen are converted with yields as shown on the diagram.

Representative tests on Hydrocol gasoline blend are shown below.

Gravity, ° API	66.4
CFRM Octane, Clear	79.2
CFRR Octane, Clear	90.9
RVP at 100° F.	9.2

This brief description and accompanying flow diagram of the Hydrocol Process was made possible through the courtesy of Texaco Development Corporation, 135 East 42nd Street, New York 17, New York, licensors of the Hydrocol Process.



THE HYDROCOL PROCESS

COURTESY, TEXACO DEVELOPMENT CORPORATION

Hydroforming Process

THE M. W. KELLOGG COMPANY

HYDROFORMING is primarily a catalytic process for converting a low-octane naphtha into a high-octane gasoline suitable for fuel in high-compression automobile engines. This conversion or reforming is accomplished largely by means of dehydrogenation and cyclization reactions. These reactions are promoted by molybdena-on-alumina catalyst at temperatures of 900-1000° F. and pressures of 150-300 psig with a high partial pressure of hydrogen in the reaction zone. The hydrogen partial pressure is maintained by recycling hydrogen-rich gas to the reaction zone along with the vaporized fresh feed. The process usually produces a large amount of net hydrogen, which makes possible the hydrogen-rich recycle.

Naphthenes (cycloparaffins) in the feed stock are dehydrogenated, and some molecules are slightly rearranged to form aromatics. Considerable cyclization of straight-chain hydrocarbons also takes place. If any olefins are present, as is the case when same cracked naphtha is included in the feed, these are almost completely converted to paraffins or ring compounds. Over 90 percent of the sulfur contained in the naphtha feed is removed, giving a low-sulfur reformate even when a sulfur-bearing naphtha is charged. Additional characteristics of hydroformer gasolines are low Reid vapor pressures; high clear octane numbers; high lead susceptibilities.

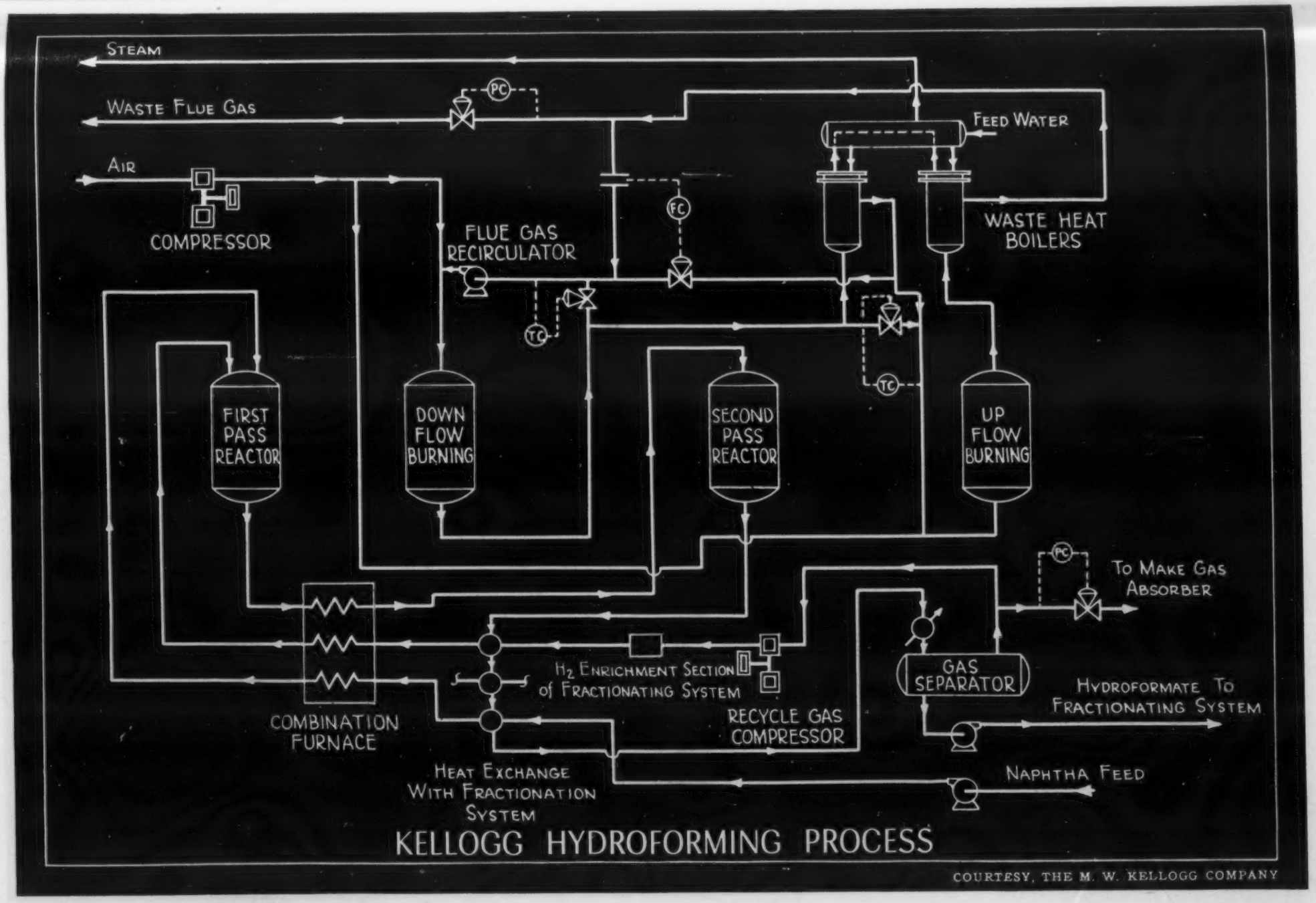
Yields of 78-80 volume percent of 80 CFR-M octane, 100 percent C_4 recovery gasoline are obtained, with the balance of the material going approximately three fourths to C_4-free gas and one fourth to polymer and carbon. When operating to lower octane levels it is possible by blending in extraneous butanes to the 10-pound Reid vapor pressure level, to obtain a yield based on feed of 100 percent of 69-octane gasoline which can be brought up to 80 CFR-M octane by the addition of 1.5 cc. of TEL.

Hydroforming is carried out in a unit usually having four or more reactors filled with catalyst, half of which are always in series on reaction, and the other half in different stages of regeneration. The reactors are switched from reaction through regeneration and back to reaction in cycles of 8 to 16 hours duration by an electric cycle-controller. Regeneration is necessary because a small deposit of carbon and a partial reduction of the metal in the catalyst combine over a period of hours to reduce the activity of the catalyst. The carbon is burned and the catalyst reoxidized by combustion in a stream of air greatly diluted by cooled and recirculated flue gas.

The reaction products are recovered and fractionated into: (1) a hydrogen-containing gas substantially free of hydrocarbons heavier than propane, (2) a highly aromatic "polymer" boiling above 400° F., and (3) a depropanized, 400° F. end-point gasoline.

The following table shows the inspections of a typical naphtha feedstock together with the inspections of the gasoline produced from it.

	FEED West Texas and Mid-Continent Mixed Naphtha	Hydroformer Gasoline
Gravity, °API	50.5	50.5
ASTM, Distillation, °F:		
IBP	174	104
10 Percent	236	156
50 Percent	311	258
90 Percent	384	338
EP	442	399
Octane Number, CFR-M clear	39.7	80.3
Aniline Point, °F	124	57
Bromine Number	2.2	3.2
Lamp Sulfur, Weight Percent	0.220	0.015
Yields, Vol. Percent of Feed (Output Basis):		
C_4-Free Gasoline	73.6	
Butanes	10.6	
Polymer	3.2	
Yields, Wt. Percent of Feed Output Basis:		
C_4-Free Gasoline	76.0	
Butanes	7.8	
Polymer	3.9	
C_4-Free Gas	10.7	
Carbon	1.6	
Total	100.0	



KELLOGG HYDROFORMING PROCESS

COURTESY, THE M. W. KELLOGG COMPANY

Hydrogenation Process

STANDARD OIL DEVELOPMENT COMPANY

HIGH-PRESSURE hydrogenation for oil processing and the methane-steam "tube" method for manufacture of hydrogen used in hydrogenation were developed in the United States by Standard Oil Development Company. Plants carrying out this process have been operated at Baton Rouge, Louisiana, Bayway, New Jersey, Port Arthur, Texas, Richmond, California, and Whiting, Indiana, as well as at various locations abroad. In addition, "tube" manufacture of hydrogen has been adapted to a number of relatively small installations, while over 100 million cubic feet per day of "tube" hydrogen capacity was installed in government plants for ammonia production during the war.

High-pressure hydrogenation has been used in manufacture of aviation base stock and motor gasoline from gasoils, high-grade diesel fuels from low-quality gasoils, water-white paraffinic kerosines from inferior-quality distillates, high-viscosity-index lubricating oils from poor-quality lubricating distillates, and also lends itself to preparation of high solvency naphthas, high-octane-number aviation safety fuels, etc. During the war much isooctane aviation blending agent was produced using high-pressure hydrogenation; an unusual adaptation was its use to make aromatic amine anti-knock addition agent for aviation gasoline from the corresponding nitro compound. High-pressure hydrogenation plants can be used for desulfurization of high-sulfur fuels, and for saturation of catalytically cracked cycle stocks to enable their recycle cracking to ultimate high production of catalytic gasoline.

In the hydrogenation process the charging stock, together with sufficient hydrogen, is pumped into a reaction vessel containing catalyst where, at the desired temperature and at about 3000 psi, the reaction takes place. The "tube" method for hydrogen manufacture and the hydrogenation step proper are outlined in the accompanying flow sheet.

Hydrogen may be produced by any of the usual processes. Since the hydrogen does not need to be sulfur-free, its production from coal or coke by the water-gas process is facilitated; it may also be produced by liquefaction and distillation of coke-oven gases, or electrolytically, when the economics permit. But for the oil industry with its generous supply of refinery and natural gas, hydrogen is advantageously made by the treatment of hydrocarbons with steam in accordance with the following reactions:

$$CH_4 + H_2O = CO + 3H_2$$

$$CO + H_2O = CO_2 + H_2$$

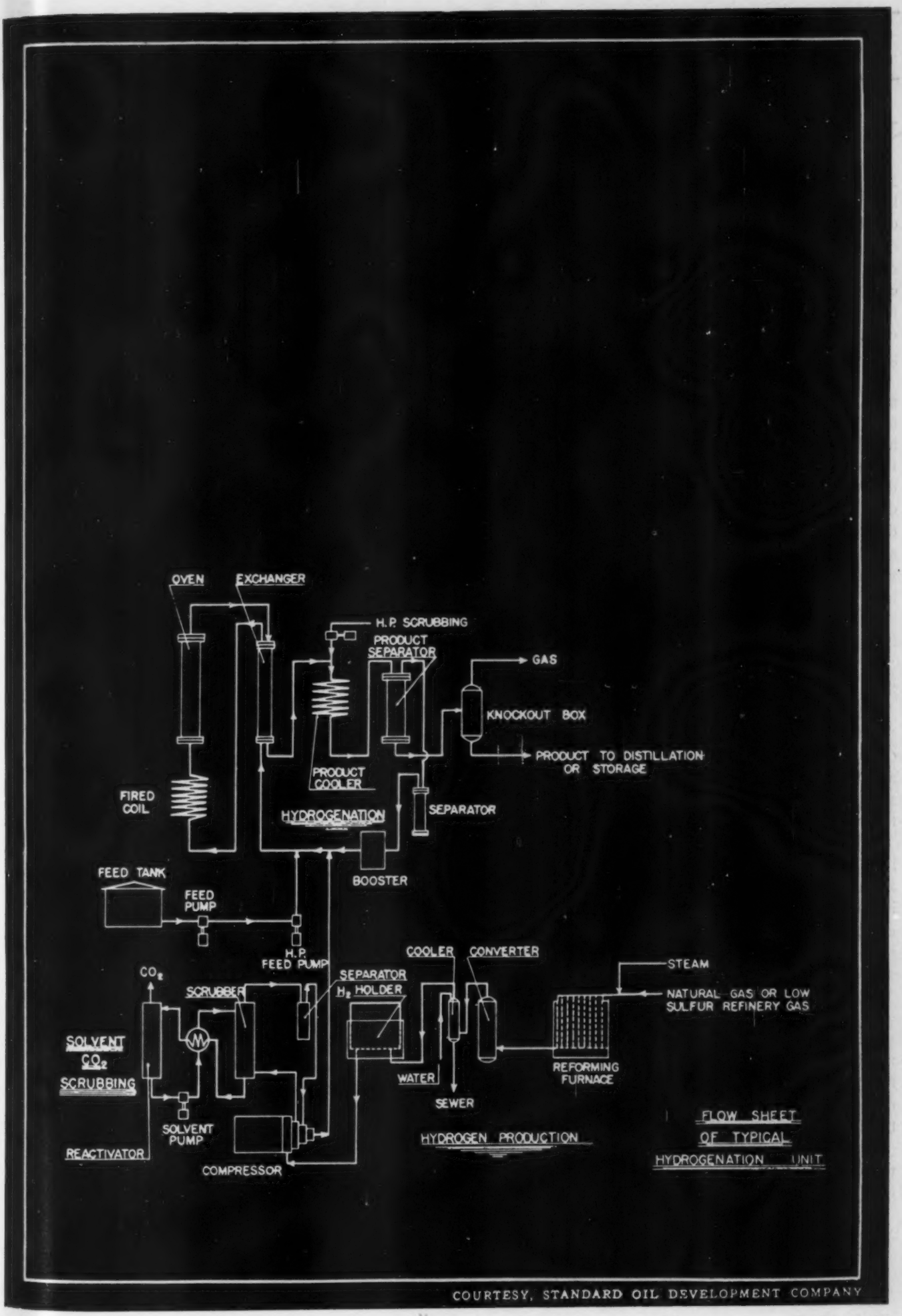
The first step is carried out in a reforming furnace, using catalyst in tubes through which reaction heat is supplied. The second, "conversion," step also uses catalyst, but does not require supply of heat. After cooling, the carbon dioxide is scrubbed out, leaving hydrogen of about 95 percent purity.

The oil and previously compressed hydrogen are mixed and delivered through heat exchangers to a coil furnace and thence to a reaction vessel containing catalyst. The degree of hydrogenation is controlled carefully by alteration of operating conditions. From the reaction chamber the products and gases pass through the heat exchangers and coolers to a high-pressure separator where the liquid product is separated from the unconsumed hydrogen and other gases. The liquid product is reduced to atmospheric pressure and sent to storage.

Gas formed in the process ordinarily is removed in the liquid product owing to its solubility at the high pressure. Such gases as are not removed in this manner may, if necessary, be taken out by scrubbing the recycle hydrogen with oil under pressure. The unconsumed hydrogen separated under high pressure from the liquid product is recycled by boosting back to full operating pressure in a booster compressor, which operates through a pressure interval equal to the pressure drop in the system. Fresh hydrogen is added to the recycle gas to make up for that consumed in process.

No coke is formed in high-pressure hydrogenation and the catalysts employed unusually are rugged, so that the process is continuous. Runs of more than a years' duration have been made on full-scale units, and total catalyst life of several years has been obtained. Hydrogen manufacture catalysts are also rugged and regularly maintain satisfactory activity for a number of years, so that only occasional small make-up is required. As a result, both hydrogenation and hydrogen manufacture sections show favorable service factor, and maintenance and turnaround costs are reduced.

This description is given through the courtesy of Standard Oil Development Company, New York, New York.



COURTESY, STANDARD OIL DEVELOPMENT COMPANY

Hygirtol Process

THE GIRDLER CORPORATION

A GIRDLER Hygirtol Plant for manufacturing high-purity hydrogen from hydrocarbons and steam combines several well established steps into a unified, continuous, low-pressure process for producing hydrogen of purity equal to or better than that produced by any other commercial process. For many uses the hydrogen from a Hygirtol Plant is preferred even to that produced by electrolysis because its oxygen content is so low.

Suitable hydrocarbons such as natural gas, refinery gases, propane and others, are purified to remove sulphur compounds, and then are reacted with steam at elevated temperatures (about 1500° F.) over a nickel catalyst to convert the hydrocarbons to hydrogen, carbon monoxide, and carbon dioxide. By employing suitable catalysts in a furnace of special design, the decomposition of the hydrocarbons is substantially complete so that the hydrogen produced will contain less than 0.1 percent of residual hydrocarbon.

The following chemical reactions occur in the furnace:

$$(1) \quad C_nH_{2n+2} + nH_2O = nCO + (2n+1)\,H_2$$
$$(2) \quad C_nH_{2n+2} + 2nH_2O = nCO_2 + (3n+1)\,H_2$$

Similar reactions occur with unsaturated hydrocarbons such as ethylene and propylene. These reactions are all endothermic, so that heat must be supplied in the furnace to maintain the required temperatures.

The mixture of hydrogen, carbon monoxide, carbon dioxide, and steam from the furnace is cooled to about 700° F. by the further addition of steam and is passed into a converter containing an iron-oxide catalyst where the carbon monoxide reacts with steam to produce carbon dioxide and hydrogen as shown in the following equation:

$$(3) \quad CO + H_2O = CO_2 + H_2$$

This reaction is slightly exothermic, so no heat need be added to the converter. It is a reversible reaction, with about 90 to 95 percent of the carbon monoxide being converted to carbon dioxide under the conditions normally encountered.

The gas mixture from the converter is cooled and the carbon dioxide is removed by the Girbotol Process. The hydrogen is then purified further as required to remove the residual carbon monoxide (about 1 percent) by further carbon monoxide conversion steps followed by the removal of carbon dioxide by the Girbotol Process, or by methanation of the residual carbon monoxide according to the following reaction:

$$(4) \quad CO + 3H_2 = CH_4 + H_2O$$

This reaction is exothermic and is carried out at slightly elevated temperatures in the presence of a catalyst.

The flow diagram on the opposite page illustrates a Hygirtol plant producing high-purity hydrogen using propane as process material and fuel. A typical analysis of the hydrogen produced in a plant of this type is as follows:

Analysis of Hygirtol Plant Hydrogen

Component	Percent
Carbon dioxide	0.01
Carbon monoxide	0.00
Methane	0.10
Nitrogen	0.01
Oxygen	0.00
Hydrogen	99.88
	Total 100.00

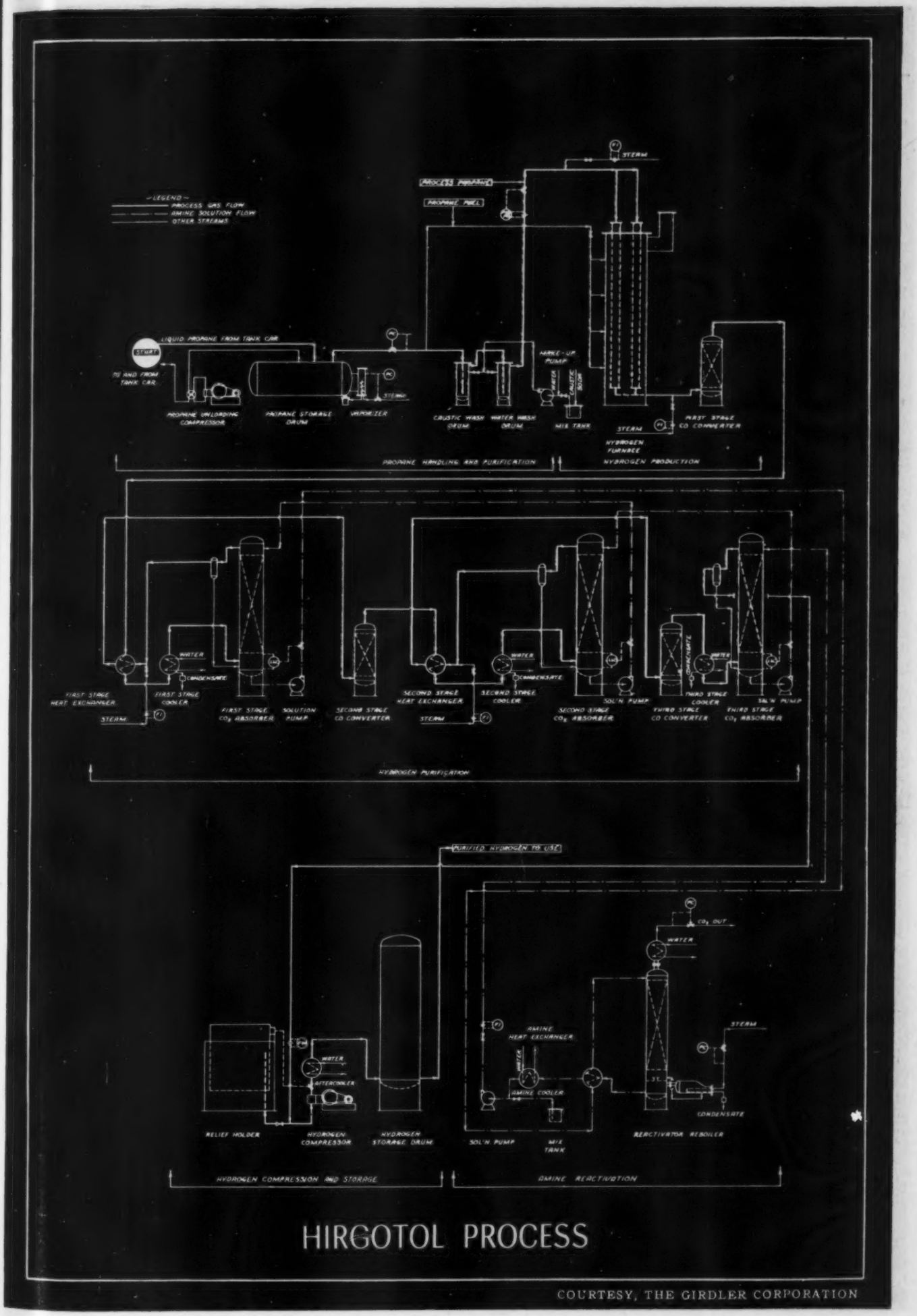
The material and utility requirements for producing 1000 cubic feet of high-purity hydrogen in this type of plant are given below:

Material and Utility Requirements per 1000 SCF of Hydrogen

Material or Utility	Quantity
Process Material:	
Propane, gallons	2.75
Or Natural gas, cubic feet	250
Fuel (gas or oil) BTU	250,000
Steam pounds	380
Cooling water, gallons (30° F. rise)	1600
Power, KWH	2
Chemicals, cost in cents	2

The Hygirtol process is continuous, and it can be made completely automatic in operation. The catalysts used are rugged and long-lived, so that replacement costs amount to only one or two cents per thousand cubic feet of hydrogen produced. Only one operator is required per shift, and maintenance is very small.

This information is presented through the courtesy of The Girdler Corporation, Louisville, Kentucky, owner and licensor of the Hygirtol Process.



HIRGOTOL PROCESS

COURTESY, THE GIRDLER CORPORATION

Hypersorption Process

UNION OIL COMPANY OF CALIFORNIA

HYPERSORPTION is a process for the separation of hydrogen, light hydrocarbons and other gaseous compounds which are not readily or economically handled by conventional fractionation procedures. The process employs a moving bed of activated carbon granules and depends on the ability of this material selectively to adsorb heavier hydrocarbons or larger molecules to a greater degree than light hydrocarbons or smaller molecules.

Referring to the flow diagram, it will be noted that the Hypersorption unit, termed a Hypersorber, has three major sections: a cooling section at the top, an adsorption and rectifying section in the middle, and a steam stripping section at the bottom. An elevator or gas lift is employed to convey the granular charcoal from the bottom of the unit to the top in a closed cycle of circulation.

The feed gas enters through a distribution plate and passes upward counter-current to the moving bed of activated charcoal. In its passage through the bed, the heavier compounds are adsorbed and retained on the charcoal. The top product gas, comprising the lightest constituents in the feed, disengages from the charcoal below the cooler and passes off through a cyclone. The charcoal passes downward below the feed point to the rectifying section where it contacts a reflux of heavy constituents, separated in the stripper which serves to liberate lighter constituents adsorbed on the downward-moving charcoal. A concentration of these latter constituents in the vapor phase results and they can be disengaged as a side-cut product gas at a point between the feed and bottom product gas disengaging sections. As the charcoal flows past the bottom product gas disengaging section into the steaming section, it meets an upward flow of steam which liberates the adsorbed heavy components, and these flow upward either to return as reflux or pass out as bottom product gas from the Hypersorber.

The charcoal flows from the steaming section into the stripping section wherein it passes down through tubes which are heated by a circulating heat-transfer medium and through which steam is passed counter-current to the charcoal. The hottest point in the stripper is at the bottom of this section and substantially all residual adsorbed material is removed during the passage of the charcoal through this zone.

The flow of charcoal leaving the stripper is controlled by means of a specially designed reciprocating feeding mechanism which drops the charcoal into a sealing leg in which a level is maintained to restrict the flow of steam into the charcoal lift.

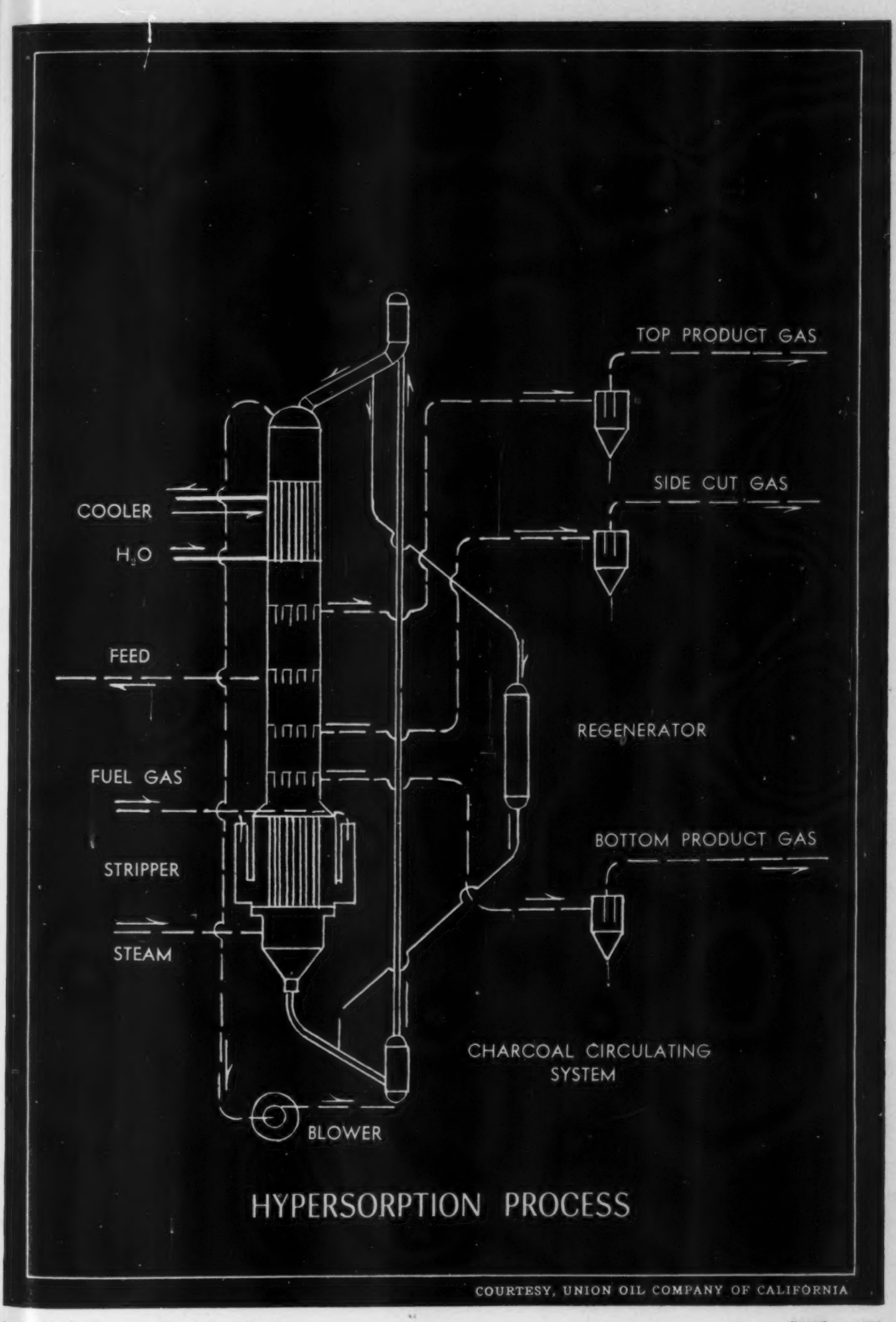
A charcoal regeneration unit is provided to act in parallel with the Hypersorber for the purpose of carrying out high-temperature steam treatment of the charcoal to maintain its activity. If such regeneration were not provided, the activity would slowly drop off due to the accumulation of heavy residual compounds on the charcoal.

The charcoal is transported to the top of the tower by means of an elevator or gas-lift system and is dropped into the reserve charcoal hopper at the top of the unit. The hot charcoal passes down from the hopper into the cooling section consisting of a tube-and-shell exchanger wherein the charcoal is cooled to the desired temperature for adsorption.

A part of the top product gas stream from the Hypersorber passes up through the cooler and hot charcoal hopper serving to remove any residual water and steam remaining on the charcoal from the stripping operation.

Important to the operation of a Hypersorption unit is the automatic temperature controller employed to maintain a close separation of the components in the unit. A substantial temperature break exists in the adsorber corresponding to the variation in the gas composition in the Hypersorber bed. By controlling this break to a point somewhat below the side-cut disengaging section it is possible to maintain a very close control of the separation process. The temperature controller operates the make-gas control valve which determines the flow of the stripped components to production or reflux. A similar setup controls the side-cut production and purity.

Among the applications for the Hypersorption process are the separation of hydrogen from methane in hydrogenation and hydroforming plants and for the separation of the ethylene and ethane from hydrogen and methane as well as the heavier constituents of the gas streams in ethylene plants. In this latter application the Hypersorber, among other things, takes the place of the so-called high-pressure, low-temperature demethanizer in the conventional ethylene plant. Hypersorption also is useful for recovery of helium, fractionation of natural gas, purification of carbon dioxide, etc. Detailed information on specific problems can be obtained from Union Oil Company of California, Los Angeles.



HYPERSORPTION PROCESS

COURTESY, UNION OIL COMPANY OF CALIFORNIA

Indiana Isoforming Process

STANDARD OIL COMPANY (INDIANA)

THE Indiana Isoforming Process is suitable for the improvement of thermally-cracked naphthas, especially those of high olefin content. The hydrocarbon vapors are passed over solid catalysts at high space velocities. Although the improvement in octane rating is moderate, extremely high yields are obtained.

The degree of improvement in octane number in the Isoforming Process depends primarily upon the olefin content of the stock and the octane number level of the olefins. A high olefin content and low octane number are conducive to greater octane number improvements. A typical improvement for a Mid-Continent thermally cracked naphtha is 4 to 5 units, for example, 68 to 72 ASTM clear. Coke-still naphthas which have a higher olefin content and lower octane number can be improved 8 to 10 units, for example, from 59 to 68 ASTM clear. The process yields about 97.5 to 98.5 volume-percent liquid product based on charge. Small losses to dry gas and carbonaceous residues amount to about 1.8 percent based on volume charged. About 2.5 percent of a polymer boiling above the end point of the charging stock is formed. In addition to octane number improvement, there is an appreciable desulfurization, which improves the lead response.

The process is carried out at relatively low pressures, for example, 10 to 20 pounds per square inch, at temperatures of about 950° F. Space velocities of from 75 to 125 barrels per hour per ton of catalyst can be used. In some instances, even higher space velocities are practicable. The process makes use of solid catalysts and fixed-bed operation. The activity of the catalyst is maintained for a period of about 48 hours, after which the catalyst requires regeneration. It is regenerated by burning off carbonaceous deposits with a flue gas containing an average of 5 mol-percent of oxygen. The maximum regeneration temperature is 1050° F. In order that the process be continuous, it is necessary to have two reaction chambers operating in parallel, one being regenerated, while the other is on stream.

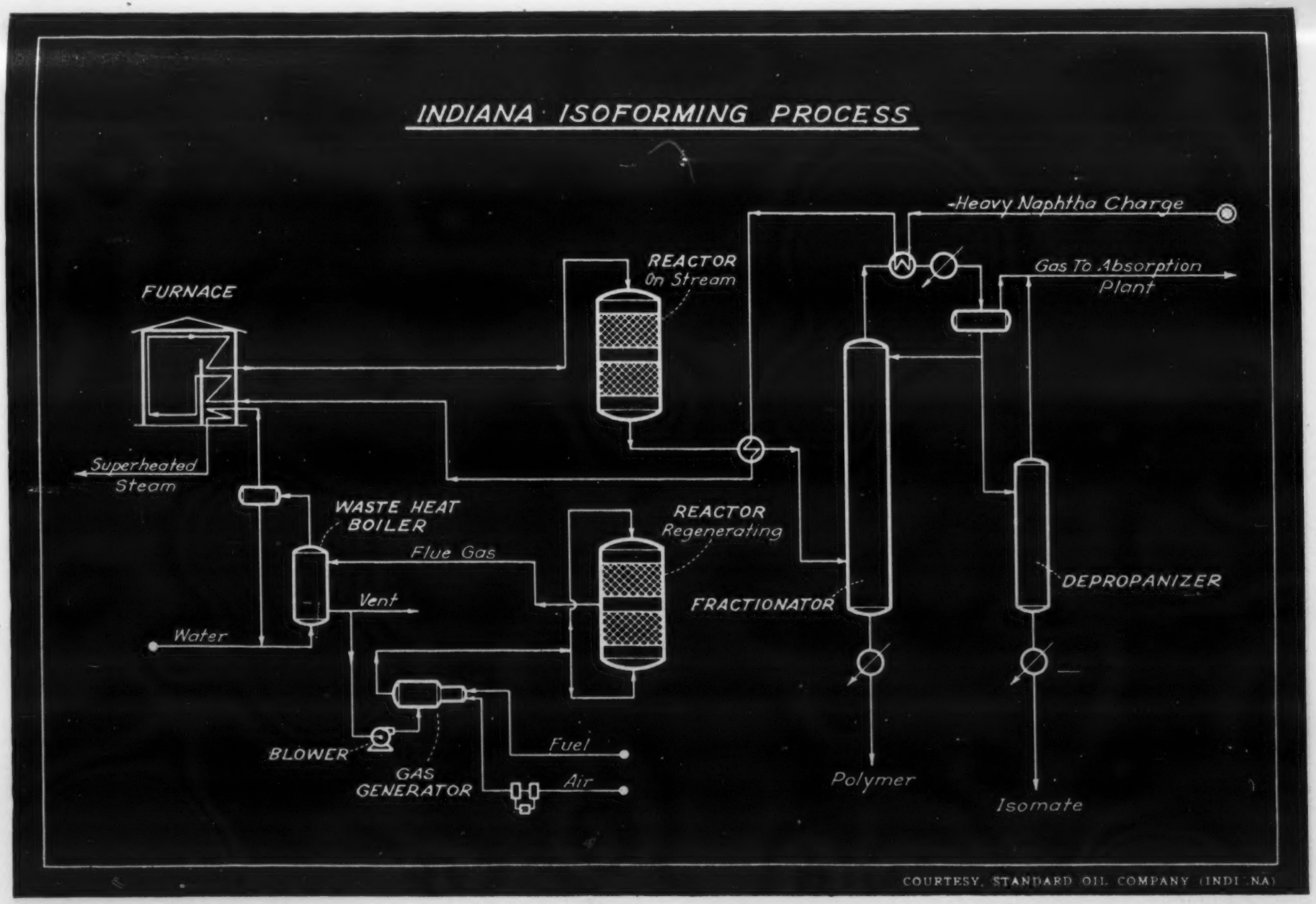
Several types of catalyst are suitable. Acid-treated clays can be used as can synthetic catalysts of the silica-alumina type. Synthetic zeolites are especially effective and are preferred.

The flow diagram on the opposite page illustrates the operation. The heavy naphtha charge is heated first by heat exchange and then by a furnace to the desired temperature. It passes as a vapor into the reactor that is on stream. The "isoformate" and small traces of polymer and gas pass from the reactor to the fractionator. Polymer is removed as fractionator-bottom product; the overhead consisting of dry gas and "isoformate" passes to a separator, the gas being vented to an absorption plant and the liquid condensate passing to a depropanizer. The "isoformate" product is removed as bottoms from the depropanizer.

A gas generator operating on air and fuel gas provides a flue gas containing about 5 percent oxygen, which is used for regeneration in the other reaction chamber. The effluent gas stream from the regeneration passes to a waste-heat boiler for heat recovery, and thence to the stack.

Isoforming units have been designed to process 20,000 barrels per stream day of heavy cracked naphtha. No commercial installations have been made, since commercialization was interrupted by the war. It is only now that reconsideration is being given to their installation. The Isoforming Process should perform an important function in small refineries which have not yet installed catalytic cracking, in addition to being useful in all refineries where large volumes of coke-still naphthas and other thermally-cracked stocks are available.

Licenses for this process are available from Standard Oil Company (Indiana).


INDIANA ISOFORMING PROCESS
Heavy Naphtha Charge
Gas To Absorption Plant
REACTOR On Stream
FURNACE
Superheated Steam
WASTE HEAT BOILER
Flue Gas
Vent
Water
REACTOR Regenerating
FRACTIONATOR
DEPROPANIZER
BLOWER
GAS GENERATOR
Fuel
Air
Polymer
Isomate
COURTESY, STANDARD OIL COMPANY (INDIANA)

Indiana Isomate Process

STANDARD OIL COMPANY (INDIANA)

THE Indiana Isomate Process is a method for converting low-octane-number pentanes and hexanes into isomers of higher octane number. Liquid aluminum chloride-hydrocarbon complex promoted with anhydrous hydrogen chloride is used as a catalyst. Hydrogen is added to repress cracking and disproportionation and to maintain high catalyst activity. Conversion is accomplished by passing the liquid hydrocarbon charge in a dispersed phase upwardly through the catalyst complex. The preferred operating conditions are 240-250° F. and 700-800 pounds per square inch. Suitable charging stocks for the Isomate Process are light, straight-run naphthas containing pentanes and hexanes and but little heavier hydrocarbons.

In addition to handling mixtures of pentanes and hexanes, the process is applicable for the isomerization either of pentanes alone or hexanes alone if the refinery situation indicates such operations to be desirable. However, for the isomerization of pentanes alone, the Indiana Pentane Isomerization Process is somewhat more economical.

When charging a mixture of pentanes and hexanes, the Isomate Process can be operated on a once-through basis to manufacture a product of 80 unleaded CFR-M octane number. Exceptionally high liquid yields, in the order of 100 volume-percent on charge, can be obtained in this type of operation. By fractionating the once-through isomate and recycling the low-octane-number pentanes and hexanes to the reaction zone, the quality of the product can be raised to 91 unleaded CFR-M octane number with but a slight sacrifice in yield. When recycling is employed with a charge stock consisting primarily of paraffinic hexanes, the principal product is neohexane.

Isomate plants of 5000 barrels and 1750 barrels per day capacity have been constructed and successfully operated at Whiting, Indiana, and Salt Lake City, Utah. Pentane recycling was employed at Whiting and hexane recycling at Salt Lake City.

Referring to the flow diagram, naphtha feed passes first through a calcium chloride dryer or other drying means, a sand filter, and thence to the top of a hydrogen chloride absorption tower. In the absorption tower hydrogen chloride is recovered from the recycle gas. A small amount of makeup hydrogen chloride is added as required at this point. The naphtha containing dissolved hydrogen chloride passes from the accumulator at the bottom of the absorption tower through a preheater to the reactor. The naphtha and gaseous hydrogen are introduced simultaneously through a distributor at the bottom of the reactor, which is an open vessel filled with aluminum chloride-hydrocarbon complex catalyst.

The hydrocarbons, being considerably lighter and substantially immiscible with the catalyst, pass upward through the catalyst. The effluent from the reactor, consisting of isomerized naphtha, hydrogen chloride, hydrogen, and a small amount of entrained catalyst, passes successively through a hot settler, a cooler and a cold settler. The catalyst is separated in the two settlers and returned to the reactor or to catalyst storage; hydrogen and hydrogen chloride are flashed off in the cold settler, and hydrogen chloride remaining in the effluent from the cold settler is removed in the hydrogen chloride stripper. The liquid product from the bottom of the hydrogen chloride stripper passes through a cooler and then successively through caustic and water washes to eliminate the last traces of catalyst and hydrogen chloride. Hydrogen chloride from the stripper is recycled to the absorber. The isomate product can be debutanized for blending into aviation fuel or blended directly into motor fuel.

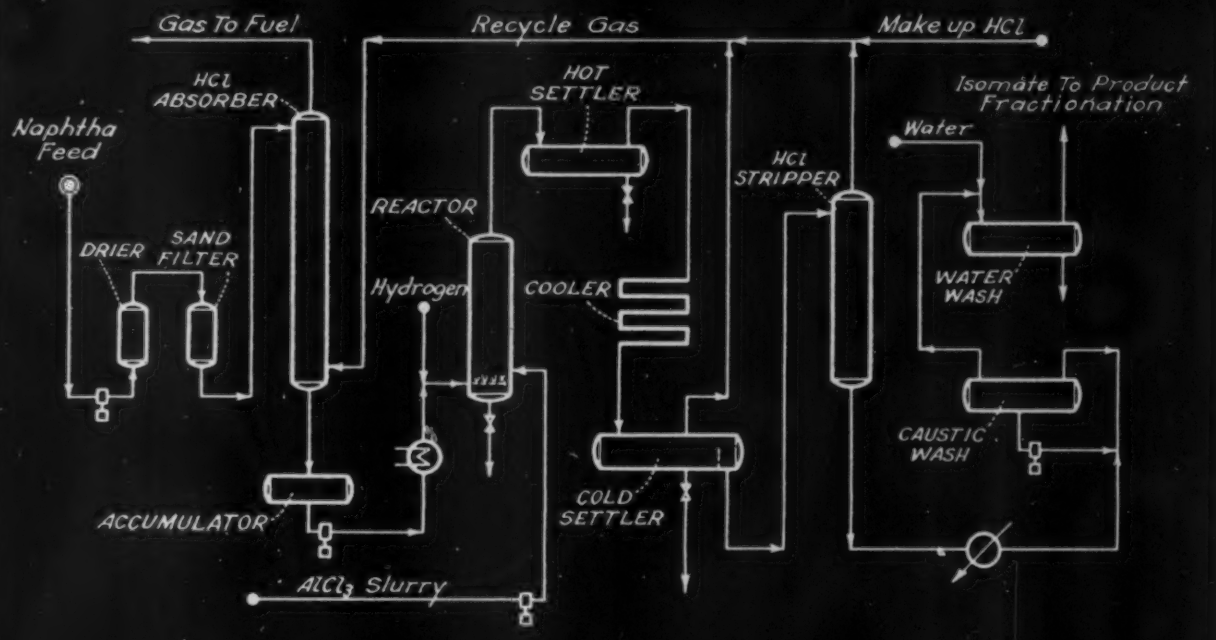
In some instances, it may be desirable to further fractionate the isomate. An isomate fractionation system is also shown on the flow diagram.

Crude isomate is passed first to a depentanizer in which butanes and pentanes are taken overhead and hexanes, along with traces of heavier materials, are removed as bottoms. The overhead butanes and pentanes are separated by fractionation, the pentanes passing to a pentane fractionator from which substantially pure isopentane is taken overhead, with normal pentane eliminated as bottoms for possible recycle to the isomate unit. The hexanes and heavier materials from the bottom of the depentanizer pass to a hexane fractionator where neohexane and diisopropyl are removed as overhead products; the bottoms are sent to a rerun tower where cyclohexane, and heavier materials unsuitable as charging stocks for the isomate process are removed. The overhead methylpentanes and normal hexane from the rerunning operation may be combined with normal pentane and recycled to the isomate unit.

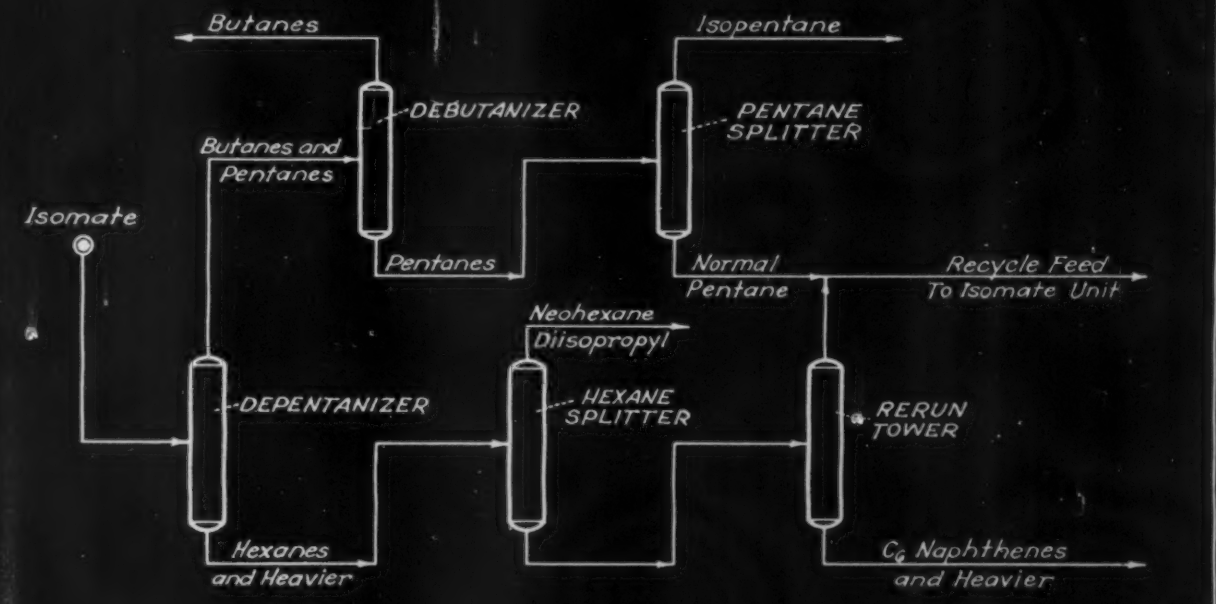
Products from the Isomate Process are primarily of interest as blending stocks for high-octane aviation fuels. However, the process is very suitable for improving the octane number of light straight-run naphthas for motor fuels where low-octane-number charging stocks such as normal pentane and normal hexane are available, or where octane ratings must be increased beyond levels attainable with existing equipment.

Licenses for operation of this process are available from Standard Oil Company (Indiana).

INDIANA ISOMATE REACTION PROCESS



INDIANA ISOMATE FRACTIONATION PROCESS



COURTESY, STANDARD OIL COMPANY (INDIANA)

Indiana Isopentane and Isobutane Processes

STANDARD OIL COMPANY (INDIANA)

THE Indiana Isopentane Process is a method for isomerizing normal pentane to isopentane. In this process, as in the Indiana Isomate Process, the catalyst is a liquid aluminum chloride-hydrocarbon complex promoted with anhydrous hydrogen chloride. In the Isopentane Process, cracking and disproportionation are suppressed by inclusion of a small amount of cyclic hydrocarbons in the feed to the isomerization unit. Although both naphthenes and aromatics are suitable for this purpose, benzene is most effective. From about 0.3 to 1.3 weight-percent based on charge is sufficient to inhibit cracking and to maintain the catalyst activity. As in the Isomate Process, conversion is accomplished by passing the hydrocarbon solution of hydrogen chloride in a dispersed phase upward through the catalyst complex. Suitable operating conditions for pentane isomerization include a reactor temperature of 195-225° F. and a reactor pressure of 260-270 pounds per square inch.

The use of benzene to replace hydrogen as a cracking inhibitor permits operation at a lower pressure and effects a considerable reduction in initial investment. The prcoess is characterized by high yields and low catalyst consumption.

An Indiana Isopentane Process installation has the added advantage that it can with little change be used for isomerization of normal butane. With such operations, a cracking inhibitor is unnecessary, since normal butane can be isomerized with negligible tendency toward cracking or disproportionation.

The Isopentane Process usually is operated on a recycle basis. An installation with a capacity of 1620 barrels per day of isopentane has been operated successfully at the Texas City refinery of the Pan American Refining Corporation.

The Isopentane unit at Texas City has been operated in conjunction with an alkylation unit, in the manner shown in the diagram. Deisobutanized alkylate is charged to a depentanizer, phere the pentanes are taken overhead and subsequently split in an isopentane tower. The feed-preparation towers also are used for product fractionation.

Hydrogen chloride from the hydrogen chloride stripper and a small amount of benzene are added to the normal pentane, and the solution is introduced through a distributor at the bottom of a column of aluminum chloride-hydrocarbon complex. The pentane isomate passing overhead from the reactor contains small amounts of entrained catalyst. A portion of this catalyst separates out in a hot settler. The effluent from the hot settler then is cooled, permitting further separation of catalyst in a cold settler. Hydrogen chloride not flashed off in the cold settler is stripped from the product in the hydrogen chloride stripper, and is recycled to the reactor. The isomerized pentane then is cooled and washed successively with caustic and water to remove the last traces of catalyst and hydrogen chloride.

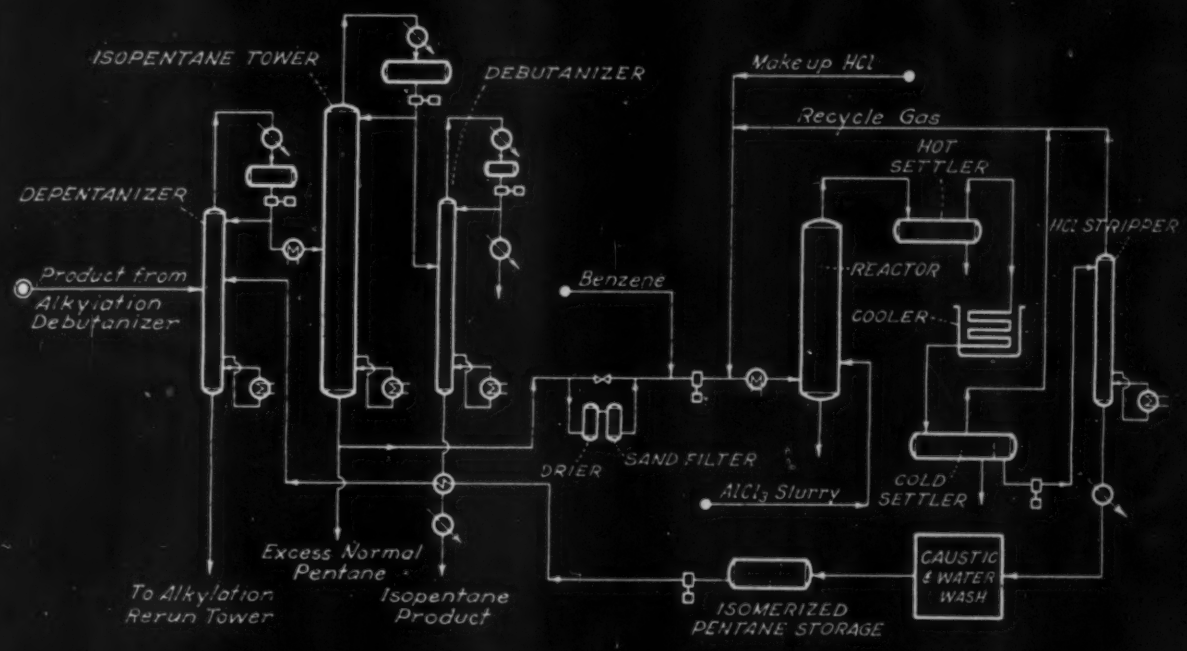
Since the equilibrium is favorable to high once-through conversion, it is not always necessary to fractionate the product. However, if nearly pure isopentane is desired, the product can be passed to the fractionation system used for feed preparation. Thus, isopentane is removed overhead from the depentanizer, debutanized, and sent to isopentane storage.

A modification of this process can be used for the isomerization of normal butane to isobutane. Butane is dried and introduced into the top of a hydrogen chloride absorption system in a manner analogous to the Isomate Process. The normal butane solution of hydrogen chloride is passed upwardly through the aluminum chloride-hydrocarbon complex catalyst in the absence of added cracking inhibitors. The temperature and pressure used in the butane isomerization process are 210° F. and 400 pounds per square inch. The isomerized butane passes through settlers for the removal of traces of catalyst and thence to a hydrogen chloride stripper. The hydrogen chloride is removed overhead and recycled to the hydrogen chloride absorber. The stripped butane is caustic-washed and passed to a debutanizer, which may be that connected with the alkylation unit operating on the isobutane produced. The butane from the debutanizer passes to a butane splitter, the isobutane going overhead to an alkylation unit or storage, and the normal butane being recycled to the isomerization system.

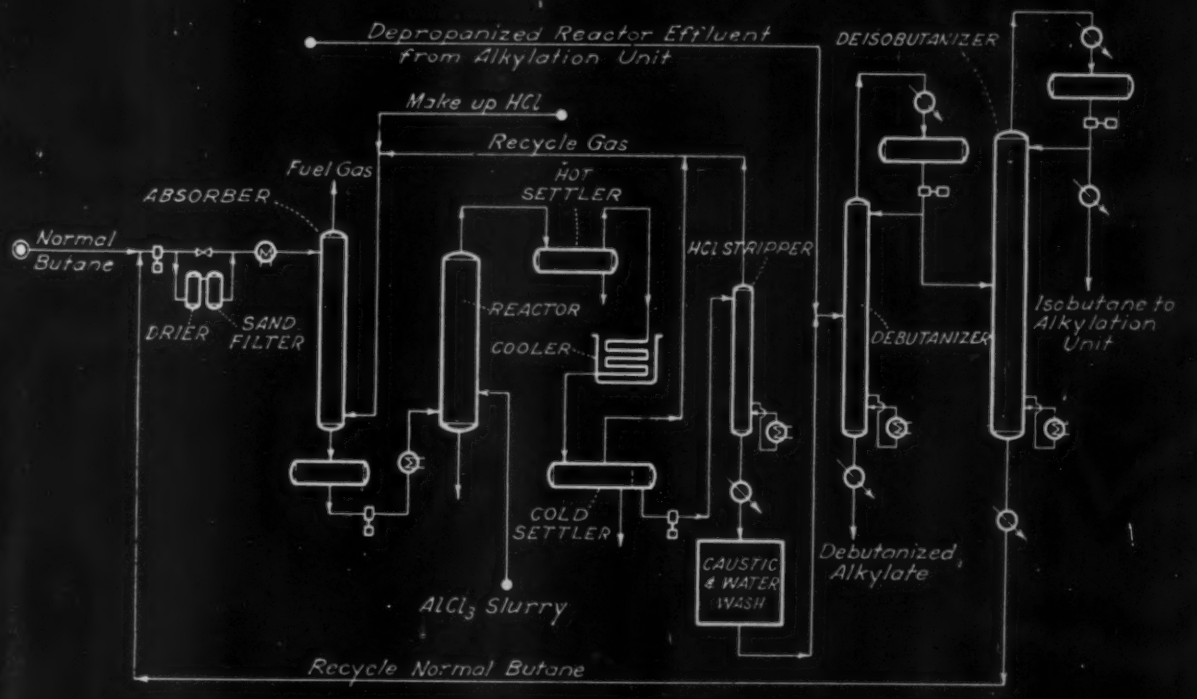
Two commercial installations of this type have been constructed and operated successfully, one at the Whiting, Indiana, refinery and the other at the Wood River, Illinois, refinery of Standard Oil Company (Indiana).

Licenses for both the butane and pentane isomerization processes are available through Standard Oil Company (Indiana).

INDIANA ISOPENTANE PROCESS



INDIANA ISOBUTANE PROCESS



COURTESY, STANDARD OIL COMPANY (INDIANA)

Light-Ends Fractionation and Stabilization

E. B. BADGER & SONS COMPANY

DEVELOPMENTS in fractionation and light-ends processing from cracking-plant products received an impetus during the war from the demands for stabilization of aviation base stocks and segregation of light ends for alkylation or other subsequent processing.

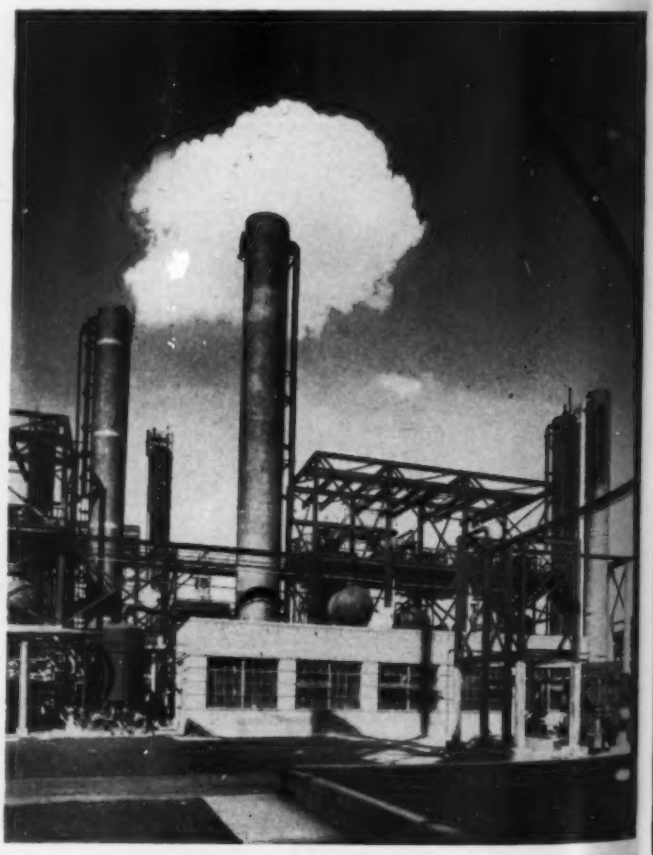
Outlined in the flow diagram is a gas plant designed and constructed by E. B. Badger & Sons Company for a large refiner in this country. This plant is designed to receive gas and gasoline from a catalytic cracking unit and from a catalytic treating unit, and to produce 30,000 barrels per day of combined liquid products as well as 6,500,000 cubic feet of fuel gas. Debutanized motor gasoline, specification aviation gasoline, commercial propane, butanes-butenes for alkylation or poly-unit feed, and isopentane are the principal end products from the unit.

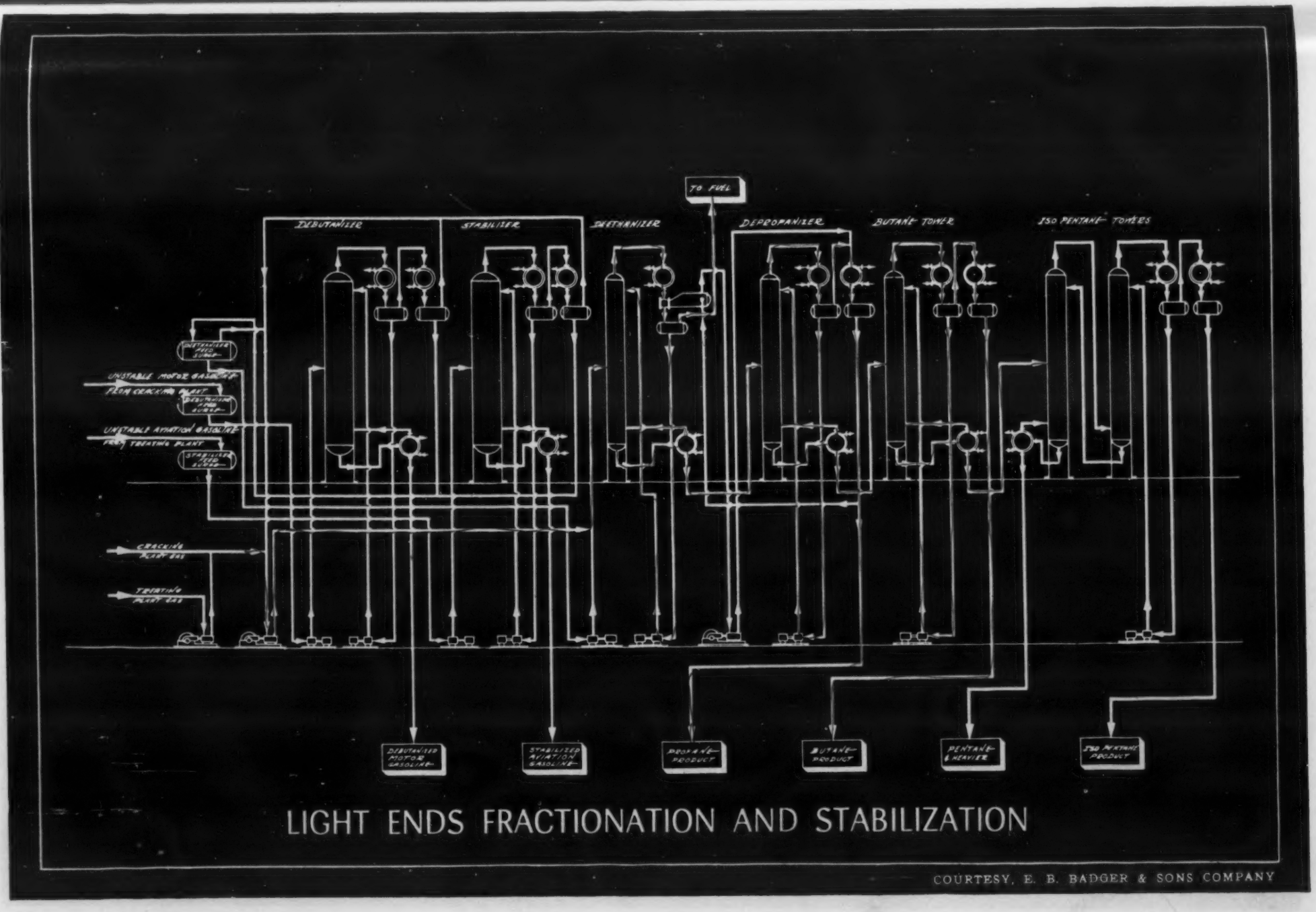
Motor gasoline from the cracking plant is charged to the debutanizer. This column is operated at 185 psig to effect recovery of the major portion of the overhead as liquid, and the debutanized gasoline goes to storage.

Aviation gasoline from the treating unit is stabilized at approximately the same pressure, also to obtain maximum recovery of the overhead as liquid, and the stabilized aviation is pumped to storage. The liquid overhead products from the debutanizer and stabilizer are pumped to a surge tank from which they are sent to the de-ethanizer.

Cracking-unit gas is transmitted to this plant at 50 psig. The smaller amount of treating-unit gas, available at lower pressure, is compressed to 50 psig, and these streams are combined with the small flow of overhead gas from the debutanizer and stabilizer, compressed in two stages and discharged directly into the de-ethanizer which operates at 450 psig. The overhead from this column at 125° F. is passed through a water-cooled condenser and then is refrigerated, providing reflux at a temperature of 45 to 50° F., the possibility of hydrate formation precluding the use of lower temperatures. A feature of the refrigeration unit design is the use of propane produced in the plant as refrigerant. The refrigeration of the de-ethanized overhead condensate is effected by the evaporation of plant propane in a kettle-type tubular unit, the propane vapor so produced being compressed and then condensed in the depropanizer overhead product condenser. Net overhead product from the de-ethanizer passes to the refinery fuel-gas system as a vapor.

De-ethanizer bottoms are delivered to the depropanizer which operates at 250 psig. In this column, commercial propane is produced overhead. The bottoms are sufficiently depropanized so that the subsequent butane product will contain less than 1 mol percent propane. The butane tower operates at 100 psig and is designed to make a sharp separation between normal butane in the overhead and isopentane in the bottoms. The bottoms from the butane tower is feed for the isopentane column. This column, operating at 22 psig, is designed to produce overhead an isopentane fraction of 98 percent purity. The bottoms from the isopentane column comprising normal pentane and heavier are pumped to storage and used in motor-gasoline blends. Although two isopentane towers are shown in the flow diagram, it will be noted that process-wise they actually constitute one tower. A split tower was used since the height of a single tower would have been excessive for the particular installation here discussed.





LIGHT ENDS FRACTIONATION AND STABILIZATION

COURTESY, E. B. BADGER & SONS COMPANY

Lubricating Oil Manufacturing System

E. B. BADGER & SONS COMPANY

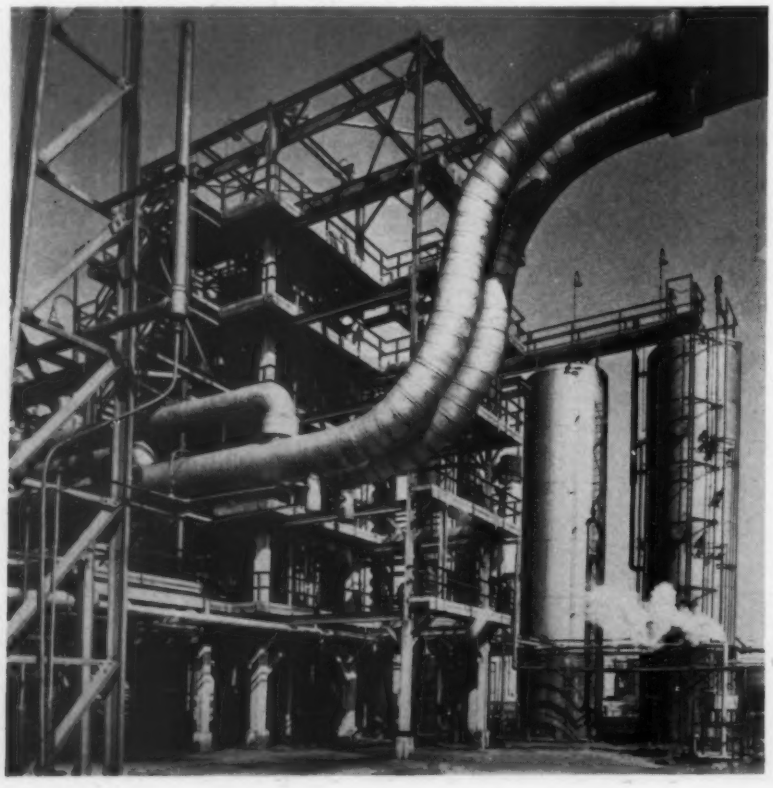
E. B. BADGER & SONS COMPANY has provided facilities for substantially all operations required for the production of high-quality lubricating oils from crudes. The steps usually required are outlined on the accompanying flow diagram and are discussed below.

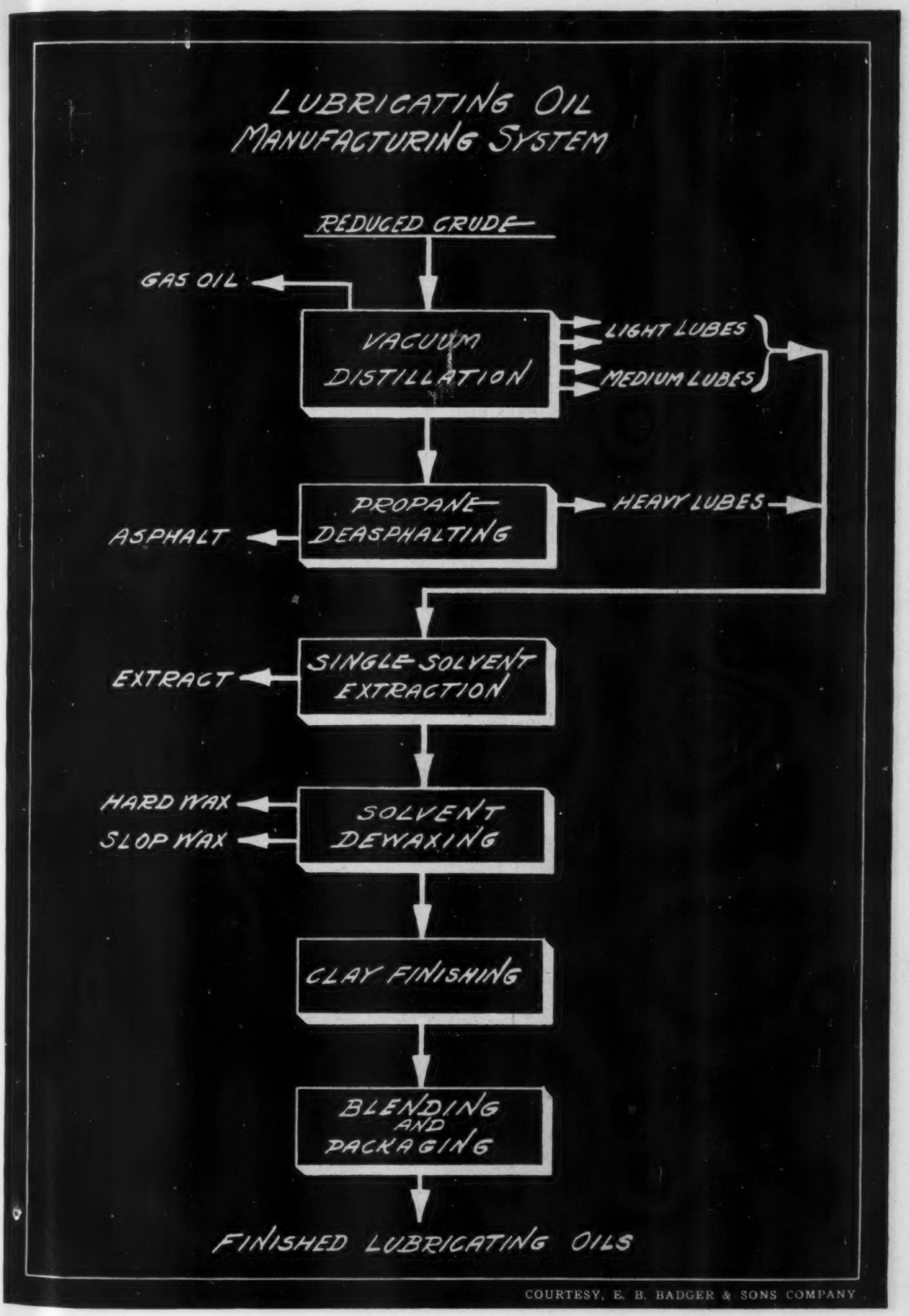
Reduced crude, from which has been removed all light straight-run products and a portion of the gasoil is charged to a vacuum-distillation unit, wherein it is fractionated into gasoil, bottoms, and the desired number of sidestream distillate lube cuts. Depending upon the crude, the operating conditions, and other factors, vacuum distillation may be the only feed-preparation step required. This is particularly likely to be true if the heavier lube fractions are only a small portion of the refiner's total demand. If a high recovery of heavy lubes is required, then with most crudes a propane-deasphalting step is recommended. By this means asphalts and resins are rejected from a propane solution of the lower-molecular-weight hydrocarbons, and a high yield of the heaviest hydrocarbons generally considered suitable for lube-oil manufacture can be obtained.

The prepared lube feed stocks are then sent (from intermediate storage) to a solvent-extraction unit wherein the low viscosity index, poor color and unstable materials in the raw lubes are removed, leaving a lube fraction which can be finished to a high-viscosity index, stable lubricating oil. Both furfural and phenol have been accorded wide industry acceptance as suitable solvents for this step. The two materials are not interchangeable, however, and due to differences in physical and chemical characteristics the plant must be designed to handle one solvent or the other. The Badger company has current experience in the design of plants to refine lube oils using each of these solvents.

The extracted lube oil, or raffinate, is then processed in a solvent-dewaxing unit. The M.E.K. process, using a mixture of methyl ethyl ketone, benzene and toluene as the solvent is widely used. The oil is mixed with solvent, chilled and filtered to remove the wax which crystallizes out under controlled conditions. A dewaxed oil of desired pour point may be produced directly, with the slack wax, if desired, being run through the unit as another charge stock from which hard wax and slop wax may be obtained; or the oil-solvent mixture may be slightly chilled, hard wax filtered out, the filtrate further chilled and filtered to remove the slop wax and to produce the dewaxed oil of desired pour•point. The choice of procedures is dictated by local conditions.

The refined, dewaxed lubricating oil streams may then be finished by percolation or by contact filtration, generally followed by brightening. The finishing step improves cast, color, stability, etc., and the oil is then ready for the addition of additives, blending to final viscosity specifications and packaging of the finished products. The Badger company has also been particularly successful in the design and installation of Filtrol Fractionation units for the combined distilling and clay contacting of lube oils. If it is desired to process a relatively wide-cut lube-oil stream through the solvent extraction and dewaxing operations, then this stream can be simultaneously fractionated and clay contacted by the Filtrol Fractionation process with attendant economies.





COURTESY, E. B. BADGER & SONS COMPANY

Lubricating Oil Processing System

THE LUMMUS COMPANY

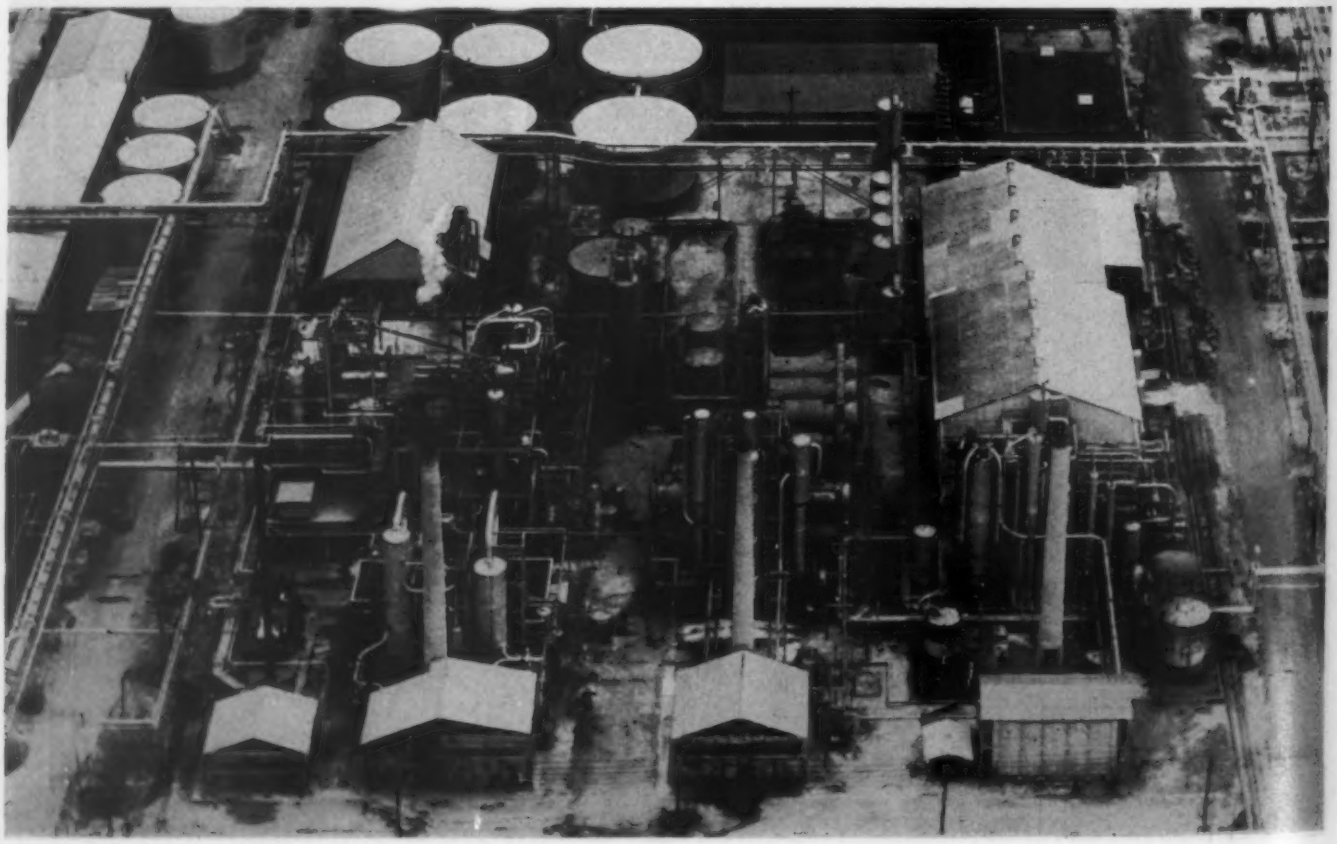
IN CONNECTION with the production of high-quality lubricating oils through the use of solvent processes for refining and dewaxing, The Lummus Company has made a number of developments towards correlating the various operations involved, with the object of coordinating the various steps so that the system as a whole will have the greatest flexibility, optimum performance, and economy of operation.

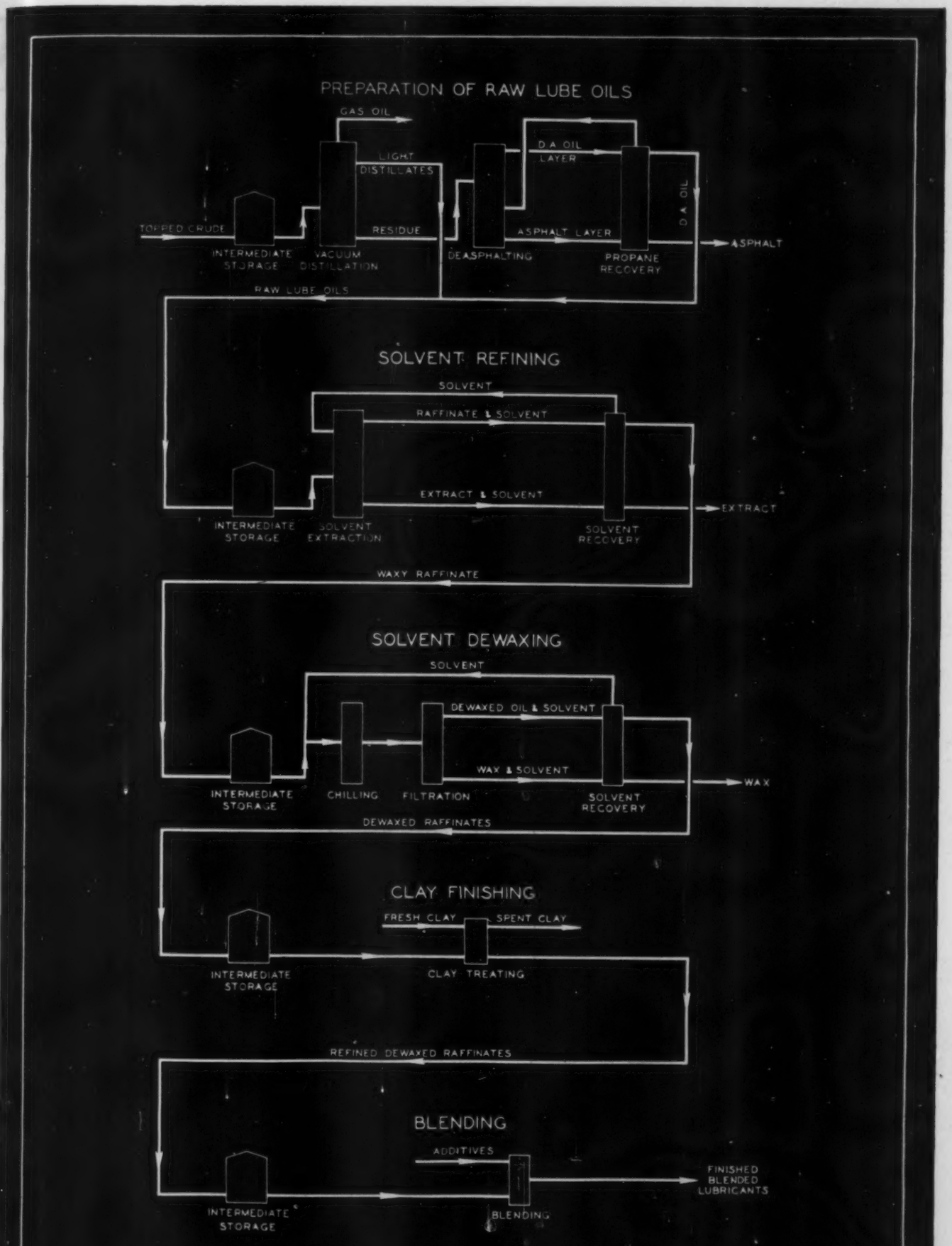
A typical processing system is shown in the diagram opposite. Topped crude is taken as the starting material and is charged to a vacuum-distillation unit which provides for the removal of the light and intermediate lube-oil distillates. The residue from vacuum distillation is charged to a propane-deasphalting unit which separates asphalt from the remaining heavy-lube distillates. With this system the maximum yield of lube oils in the topped crude is obtained. Intermediate storage is provided for the various grades so that they can be charged to the solvent-refining unit separately.

These raw lube-oil cuts are solvent refined for the removal of objectionable compounds and improvement of viscosity index. The raffinates resulting from the refining of the various grades of raw lube distillates are again sent to intermediate storage from which they are charged to the solvent-dewaxing system. A widely-used system and one with great flexibility employs a mixture of acetone or methyl ethyl ketone and benzol (this system is covered by patents owned by and licensed by Texaco Development Corporation). The essential steps in this process are (1) mixing of oil and solvent, (2) chilling of the mixture to low temperatures, (3) removal of precipitated wax by filtration, and (4) recovery of solvent from wax and from wax-free oil by distillation. The wax recovered from such a process is of excellent quality and relatively free from oil. The oil recovered has the low pourpoint demanded of high-quality lubricating oils. These wax-free oils of various grades are again sent to intermediate storage from which they are charged to the final step for finishing high-quality lubricating oils which is accomplished by the use of clay percolation or contact filtration. In this step the oil obtains the necessary treatment for obtaining the required color, neutralization number, etc. The investment for percolation plants is higher than for clay contacting while the operating costs are lower.

In view of increasingly severe requirements for lubricating-oil quality the use of additives has become generally accepted. Such materials give more desirable characteristics from the standpoint of detergency, oxidation stability and pour point. The addition of such materials is easily carried out in connection with the final blending operation.

The system described above has been incorporated in many plants over the past years for all qualities of raw-distillate stocks from a wide variety of crudes. It has great flexibility and low production costs and produces highest-quality lubricating oils.





LUMMUS COMPLETE LUBE OIL PROCESSING SYSTEM

COURTESY, THE LUMMUS COMPANY.

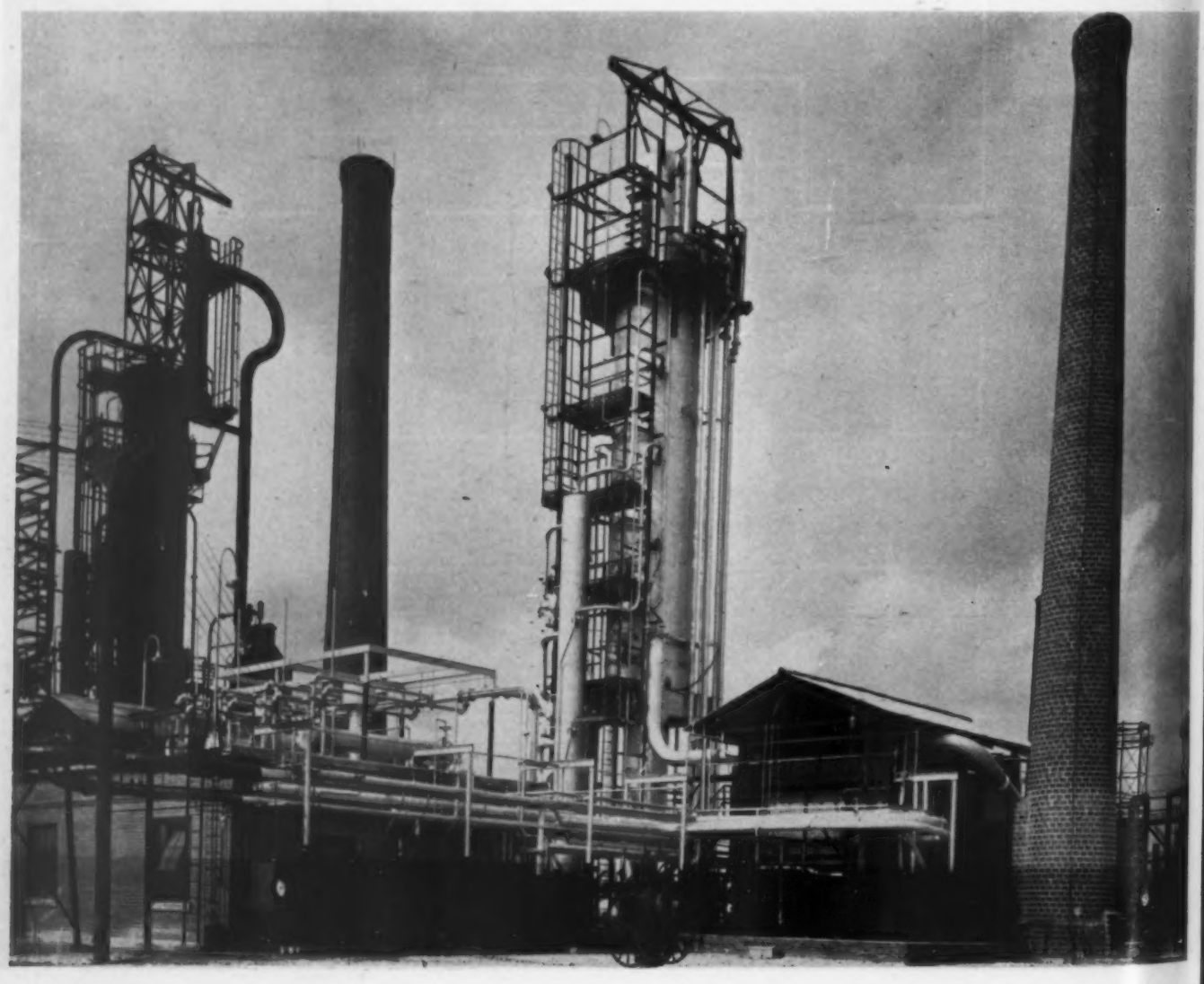
Lubricating Oil Rerun Process

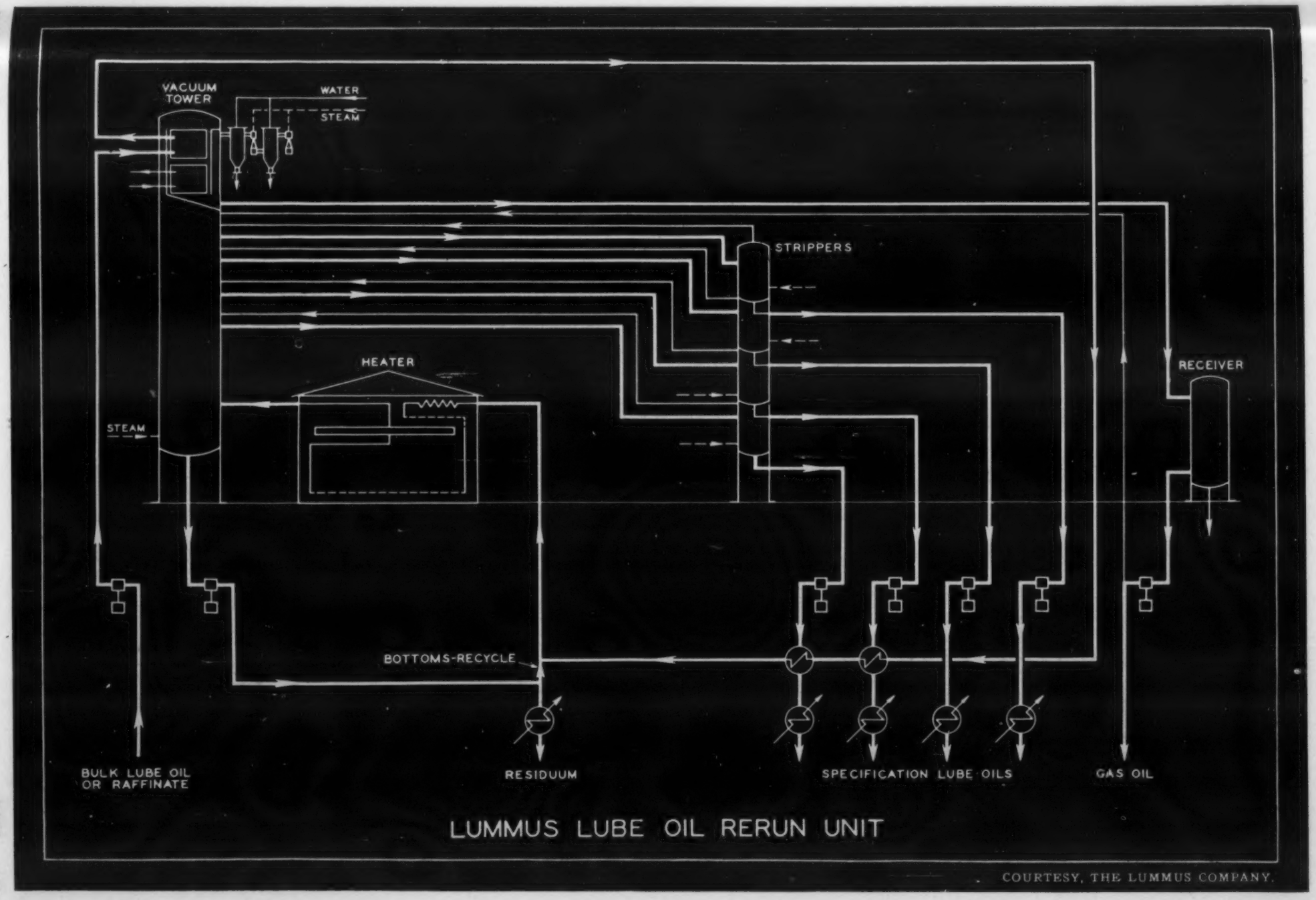
THE LUMMUS COMPANY

IN THE production of lubricating oils by modern solvent processes, there are many cases in which solvent refining can best be acomplished by operation on a bulk stock followed by redistillation of the refined bulk stock into the required viscosity grades of finished lubricating oils. To meet this need, The Lummus Company has designed and put into operation several distillation units for conducting such operations.

To insure that no deterioration of the products occurs, the distillation is carried out under vacuum and careful consideration is given to the design of fractionation and condensing equipment as well as of the oil-heating unit. The installation shown in the photograph is diagrammatically illustrated on the flow sheet opposite. The charge material is a bulk raffinate from solvent refining. The unit is designed for the production of a minimum overhead, three side streams and a minimum residue. The overhead stream is material of low viscosity which is present in the bulk material from prior operation. The amount of this light material usually is very small, since it is not economical to have any significant quantity of such material processed in a solvent refining plant. Similarly, the amount of residue is also a minimum, as this material represents chiefly the concentration of color bodies and extremely viscous fractions not completely removed by solvent refining. In view of the necessity for providing sufficient heat to carry out distillation, recirculation of the residue was provided for in such amount that this heat could be added.

The compact arrangement of equipment is clearly shown by the accompanying photograph in which one of the outstanding features is the use of the Lummus type condenser section installed within the tower shell itself. The advantages of this arrangement are obvious when savings in structures, large vapor lines, fireproofing, etc., are taken into account. It should be noted that there is no sacrifice of accessibility through the use of this arrangement. The design of this type of condenser is such that very low pressure drop and excellent liquid sub-cooling are obtained. Both of these points are of great importance in vacuum distillation units for efficient and economical operation.





LUMMUS LUBE OIL RERUN UNIT

COURTESY, THE LUMMUS COMPANY.

Naphtha and Gasoil Polyforming Process

THE LUMMUS COMPANY

THE Polyform Process, licensed under patents of Phillips Petroleum Company and Gulf Oil Corporation, affords an efficient means for converting straight-run naphthas, gasoils and extraneous light hydrocarbons into high-quality motor gasoline. Polyform gasolines characterized by high research octane numbers possess excellent road performance at all speeds, and find use as the primary component of regular- and special-grade gasolines which must meet severe competitive specifications.

A Polyform plant is a self-contained unit, designed to process naphthas, gasoils and condensible light hydrocarbons simultaneously. The unit is complete with heaters, high-pressure fractionator, stabilizer and an absorber, which equipment affords the means to condense the light hydrocarbons for the furnace charge without necessity of refrigeration. Propane-propylene and butane-butylene produced in the process are largely condensed with normal cooling water supply and the balance is absorbed in the naphtha or gasoil feeds. Extraneous light hydrocarbons may be charged into the furnace-feed accumulator as a liquid or charged to the main high-pressure absorber. Condensed liquid gas is circulated with the feed and the presence of this material in the furnace charge enables substantially more severe temperature and pressure conditions than could otherwise be achieved. This, in addition to the polymerization of the light hydrocarbons, results in the high gasoline yield and octane associated with the Polyform process. By varying the charge rate of extraneous butane-butylenes, an efficient means of plant vapor pressure balancing is afforded.

The Polyform Process has found most extensive application in the processing of low-octane naphthas, although there are in operation a number of units charging gasoils. The process is particularly well suited for the conversion of refractory catalytic cycle stocks requiring more severe conditions than can be achieved in conventional thermal-cracking plants. Yields of 10-pound RVP gasoline ranging from 60-80 percent have been obtained from virgin gasoils, while yields of 50-70 percent will be derived from catalytic cycle stocks with ASTM octane of 72-76 for virgin stocks and somewhat higher for the cycle oils.

Naptha Polyform units charging a wide variety of heavy naphthas with and without extraneous condensibles achieve higher yields of higher octane gasoline than is possible with conventional reforming units operating in conjunction with catalytic-polymerization facilities.

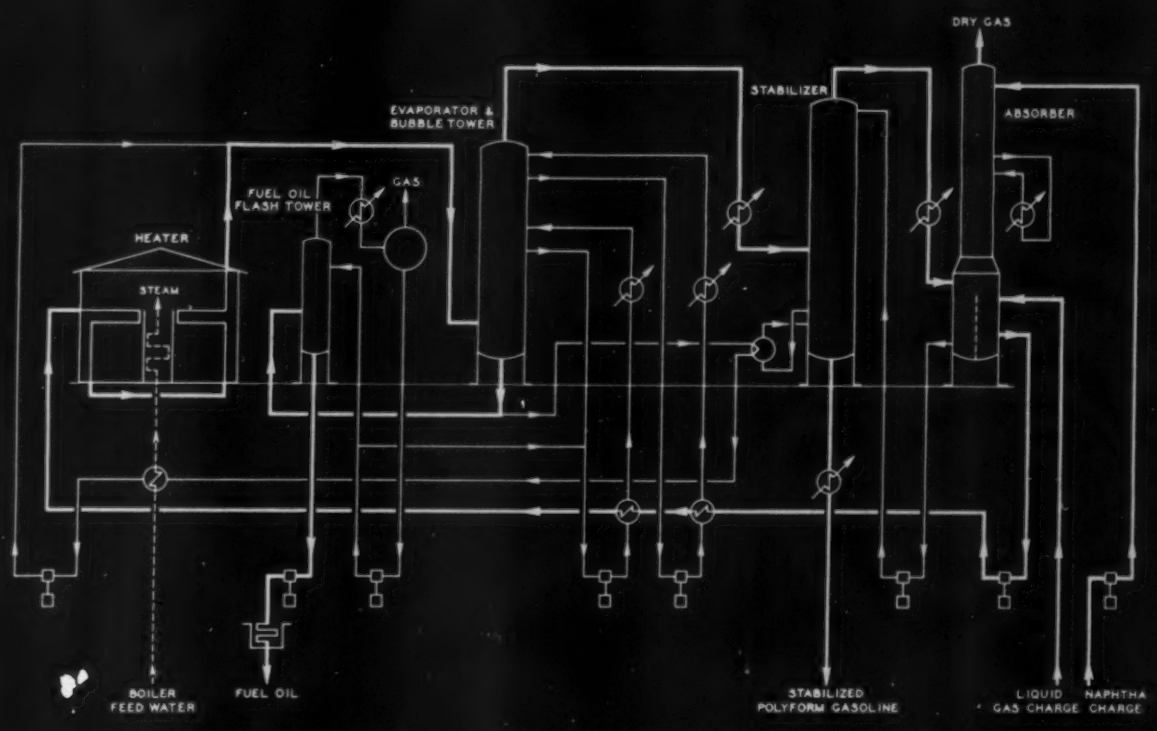
The flow sheets on the page opposite illustrate the process arrangement of typical naphtha and naphtha-gasoil polyform units. Either unit may accommodate outside gas charge as desired.

In the Naphtha Polyform unit the naphtha is charged to the absorber which is so operated that about 80-90 percent of the propane in the wet gas entering the tower is absorbed. At the base of the absorber the rich oil combines with stabilizer overhead condensate to make up the furnace feed which is charged with a high-pressure centrifugal pump to that the resultant transfer line pressure is 1200-1500 psi. Evaporator bottoms is employed to reboil the stabilizer and quench the transfer line to 650-700° F. The evaporator bubble tower operates at about 400 psi pressure. The net evaporator bottom flows to a fuel oil flash tower, the overhead of which is returned to the system, and the bottoms are delivered to fuel oil. The bubble tower is controlled with intermediate and top circulating reflux systems which preheat the furnace feed while the overhead flows through a partial condenser directly to the stabilizer, which tower operates at about 380 psig. The stabilizer may operate as a depropanizer if it is desired to produce butane-butylenes for alkylation, or may operate to produce specification-vapor-pressure gasoline with excess butane-butylene being taken overhead for conversion to gasoline. A large part of the propane-propylene and butane-butylene in the stabilizer overhead is condensed and flows to the base of the absorber, while the uncondensed wet gas flows through the absorber where it is stripped of propane and is then discharged as refinery fuel gas.

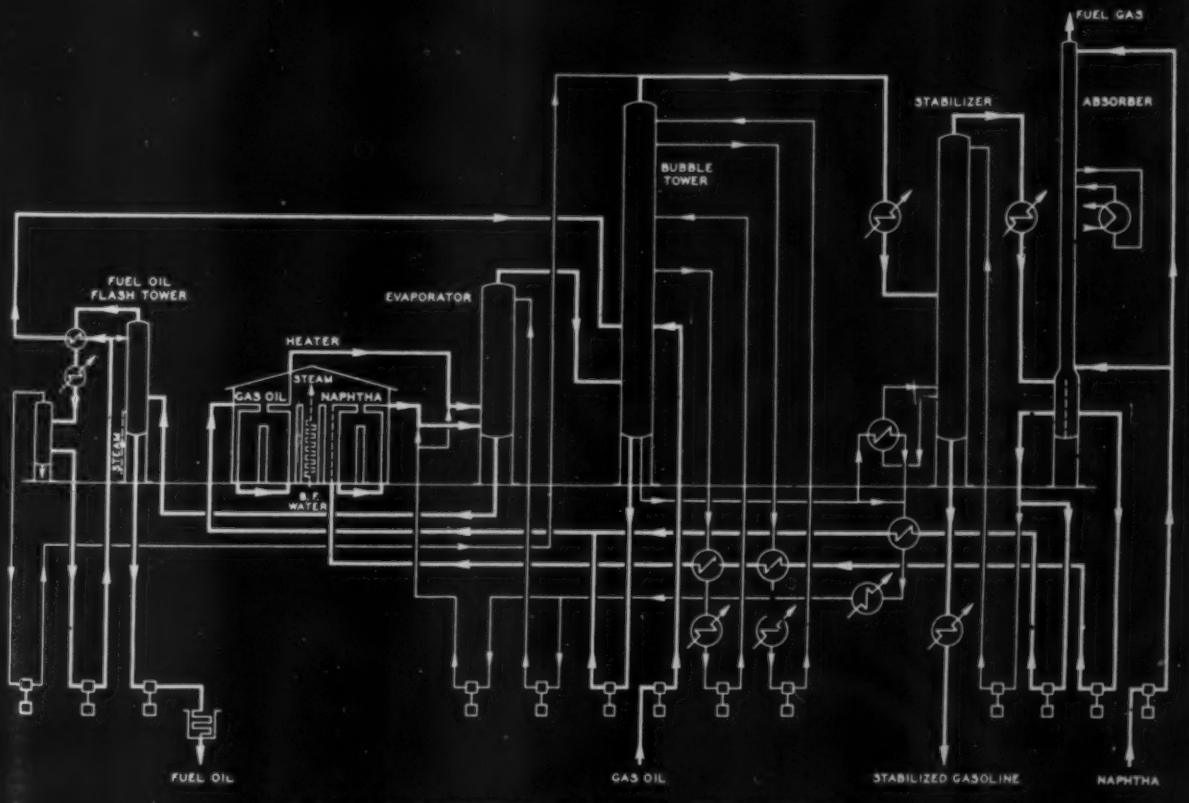
The Lummus floor-fired heater is ideally adapted for the exacting requirements of polyform service with separate heating and soaking sections. It has been found economical to use only radiant surface for the high-pressure oil service and generate steam in the convection section with forced circulation of boiler-feed water through an external steam drum. This not only brings about high fuel efficiency but provides sufficient steam for operating all of the high-pressure pumps and most of the low-pressure pumps so that the unit operates in substantially steam balance and requires no additional steam load from the refinery.

Polyform units are extremely simple and are typified by ease and stability of operation. Investment requirements for naphtha-polyform units are lower than for corresponding facilities for naphtha reforming plus catalytic or thermal polymerization.

The naphtha-gasoil polyform flow is substantially the same as for the naphtha polyform unit with only minor variations as required to accommodate the gasoil cracking. In this plant the gasoil usually is charged to the bubble tower where it mixes with the gasoil recycle to form the furnace feed. High-pressure pumps handle the gasoil furnace charge and stabilizer overhead condensate, the latter being preheated against bubble-tower circulating reflux streams before mixing with the gasoil furnace charge downstream of the high-pressure charge pumps. Naphtha-gasoil units are typified by the same ease and simplicity of operation as in the naphtha-polyform units and the number of plants in service attest to the wide acceptance of the process.



LUMMUS NAPHTHA POLYFORM OR GAS REVERSION UNIT



LUMMUS NAPHTHA-GAS OIL POLYFORM UNIT

COURTESY, THE LUMMUS COMPANY.

Natural Gasoline Refrigeration Process

SIGNAL OIL AND GAS COMPANY

THE use of refrigeration in natural-gasoline absorption plants is becoming more general as its value for increasing plant efficiency with reasonable costs is recognized. While refrigeration is justified in many new plants, especially in cases where atmospheric cooling is not too efficient, its greatest value probably lies in the increase of the absorption capacities of existing plants. The most important application is for lowering the leanoil temperatures below that possible with atmospheric cooling. This is especially true with low-pressure absorption where lower temperatures are possible without fear of hydrates forming.

In addition to lean-oil cooling, there are a number of other services that often are justified. These include the cooling of wet gas in cases where maximum refrigeration and absorption is justified. Another service that is often of importance is the cooling of the plant rundown gasoline and vapors in order to take advantage of the greater spread between vapor pressures of the hydrocarbon fractions at lower temperatures, and at the same time to control the volume of vapors that must be handled in the plant. With refrigeration available within the plan, it often is economical to use this refrigerant in the laboratory or for air conditioning.

The design and operation of a refrigeration unit both are relatively simple. There are many minor variations in the cycle used for refrigeration as well as in the design of equipment. A typical flow sheet for such a refrigeration cycle is that shown on the opposite page. It is used in one of the plants of Signal Oil and Gas Company on the Pacific Coast. For the sake of simplicity, it is shown in connection with lean-oil cooling only. In this cycle, the small variations in the volume of liquid propane that are required for normal working conditions are carried in the surge tank. The lean-oil chillers are connected directly to this surge tank in such a manner that an excess of propane refrigerant flows from the surge tank up through the chillers as a portion of the liquid vaporizes, then recycles back into the surge tank. This allows increased turbulence within the chillers, which improves heat-exchange efficiency.

A superheater is installed between the vapor leaving the chiller and the liquid entering. This exchanger not only increases the efficiency of the refrigeration cycle, but decreases the possibility of liquid condensing during cold weather, either in the suction line to the compressor or in the compressor cylinder. With a superheater, there should be no liquid in the vapor, but for added safety a scrubber is installed between the superheater and the inlet to the compressor. A small vapor-proof float and switch is placed on this scrubber to ground the magnetos on the engines and to shut down the compressors should any liquid reach this point.

The vapor then is compressed and flows through a condenser from which the liquid refrigerant drains into the accumulator. No back-pressure valves are installed, since the lowest discharge pressure are secured by allowing the condensers to hold the minimum pressure that is required for complete condensation. The vapor within the accumulator is relatively richer in ethane and methane, and this concentration offers an opportunity to vent off a relatively high percentage of these impurities which are included in the propane feed supply over a period of time. It will be found that the removal of a small amount of vapors at this point by means of a manual vent may materially decrease the compressor discharge pressure.

In the flow sheet as shown, any oil from the compressor cylinders is removed from the surge tank. This deoiler consists of a small exchanger that is operated on a by-pass from the uncooled lean oil, thereby utilizing the cooling from the refrigerant that is vaporized, as well as concentrating the oil. The volume of refrigerant fed to the deoiler is controlled by an orifice in the vapor return to the surge tank. A gauge glass on the deoiler indicates when it is necessary to drain the lube oil that accumulates. If a pure mineral oil is used, it will be found that no difficulty will arise in removing it from the system. Care should be taken to secure the feed for any deoiler at the point of greatest probable oil concentration.

This cycle is effective, easy to operate and can be installed at reasonable cost. The propane refrigerant can be made available from practically any absorption plant. Where relatively pure iso- or normal butane already is available, this material may be used with comparable results. It is felt that the use of refrigeration will become even more general in the future, especially with the increased demand for greater extraction efficiencies due to the increasing values of butanes and propane with the expanding market for these hydrocarbons for chemicals and for liquefied petroleum gases.

This description of the application of refrigeration to the recovery of natural-gasoline processing was provided by Signal Oil and Gas Company, Los Angeles, and is based on practices now functioning in its operations.

REFRIGERATION IN NATURAL GASOLINE ABSORPTION
BLEED
ACCUMULATOR
CONDENSER
AVAILABLE REFRIGERANT
FOR OTHER SERVICES
SUPERHEATER
OIL CHILLERS
SURGE TANK
DE OILER
SCRUBBER
COMPRESSOR
COURTESY, SIGNAL OIL AND GAS COMPANY

Nitrobenzene Extraction Process

THE ATLANTIC REFINING COMPANY

THE nitrobenzene extraction process for separating the high grade lubricating oils, present in either the lube distillates or the residuum of any crude, from low grade components was developed and is commercially operated by The Atlantic Refining Company, 260 South Broad Street, Philadelphia, Pennsylvania. Through the courtesy of the company this material is presented.

A crude lubricating distillate or residuum is made up of a mixture of hydrocarbons. Some of these compounds are highly paraffinic, some are of intermediate character, and some are highly naphthenic or asphaltic. The highly paraffinic compounds are practically insoluble in nitrobenzene, the asphaltic and highly naphthenic compounds are very soluble, while the compounds of intermediate character have solubilities between the two extremes. Hence, when oil is mixed with nitrobenzene the least desirable components are most readily dissolved and removed. By varying the degree of treatment, the quality of the material remaining undissolved can be controlled over a wide range.

The process is purely physical in nature since the chemical character of the various hydrocarbons remains unchanged during the separation. If the products obtained are mixed together in yield proportions, the properties of the mixture will be the same as those of the original oil. It follows that the maximum yield of an oil of given quality will be equal to the amount of oil of that quality originally present in the stock. This maximum yield is very closely approached in practice.

Nitrobenzene has a high specific gravity and a low viscosity at the temperatures employed for extraction. As a result, mixing and settling in the extractor system are rapid and complete. The solvent does not attack the ordinary materials of construction and is stable at the temperatures employed by the process. The boiling point is low enough to permit a complete separation of solvent and oil by simple evaporation and steam stripping. Nitrobenzene and water are practically immiscible and the separation of the 0.2 percent of solvent dissolved in water is very simple. At operating and storage temperatures the solvent is practically non-volatile, and accordingly solvent losses are extremely small. The supply of nitrobenzene is large and dependable, while its cost is relatively low. Eight years of experience including laboratory, semi-plant, and plant operation have shown that the material can be used without harmful physiological effects, confirming the years of experience in the manufacture of nitrobenzene in the chemical industry.

Nitrobenzene is distinctive in its high solvent power for asphaltic, naphthenic and aromatic compounds. As a result, the nitrobenzene process requires a low solvent oil ratio to produce a given yield of a given quality oil. These ratios vary from 0.5 volumes to 2 volumes of solvent per volume of oil. The importance of this property is apparent, since solvent recovery is the major cost item in solvent refining.

Essentially the process consists of two steps: contacting the charge oil with nitrobenzene in a multi-stage countercurrent extractor composed of a series of mixers and settlers; and then recovering the nitrobenzene from the raffinate and extract solutions. The raffinate solution contains the paraffinic fraction of the charge stock in which some nitrobenzene has been dissolved, while the extract solution consists of nitrobenzene and the undesirable material which it has dissolved. The nitrobenzene recovered from these solutions is re-used.

The flow of material is illustrated by the diagram on the opposite page. The charge oil is first pumped through a water-cooled chiller and thence to a brine-cooled chiller. At this point it is diluted with extract solution from the second stage of the extractor. The mixture is then cooled to the operating temperatures and enters the first settler. Fresh or previously recovered nitrobenzene flows through an exchanger and cooler to enter the extractor at the fifth stage mixer, and flows through the extractor system counter-current to the oil. The raffinate and extract solutions flow continuously from each settler into mixers or to the recovery system. The extract solution flows from extractor 5 to extractor 1 in succession, while the raffinate solution flows from 1 to 5. The extract solution leaving extractor 1 and the raffinate solution leaving extractor 5, in which it has been contacted with pure nitrobenzene, are ready for solvent recovery.

The extractor system may be operated at constant temperature (40-80°F.) throughout or with a temperature gradient, as conditions dictate. Several modifications of the extractor system have been developed and can be applied as required by the stock to be treated and the products desired.

The extract solution is heated in a series of exchangers and then delivered to the evaporators B where the nitrobenzene present is boiled off under reduced pressure, using closed steam as the heating medium. The nitrobenzene vapor is condensed for re-use. Plant operating pressures do not exceed atmospheric, and the maximum temperature used is 350°F. The extract leaving the evaporators contains about 1 percent nitrobenzene which is removed by stripping with open steam in tower C. The raffinate solution is treated in a similar fashion in the evaporators D and stripper E.

Any water which has been in contact with nitrobenzene in the process is collected in settler F. The nitrobenzene which separates is returned to storage or sent to evaporator B for redistillation. The aqueous layer from settler F is heated by exhaust steam to about 125°F. and about 15 percent of the water is vaporized under vacuum in evaporator G. The vapor, which contains all of the nitrobenzene in the water charged to evaporator G, is condensed and returned to the separator. The unvaporized water is free of nitrobenzene and can be discarded.

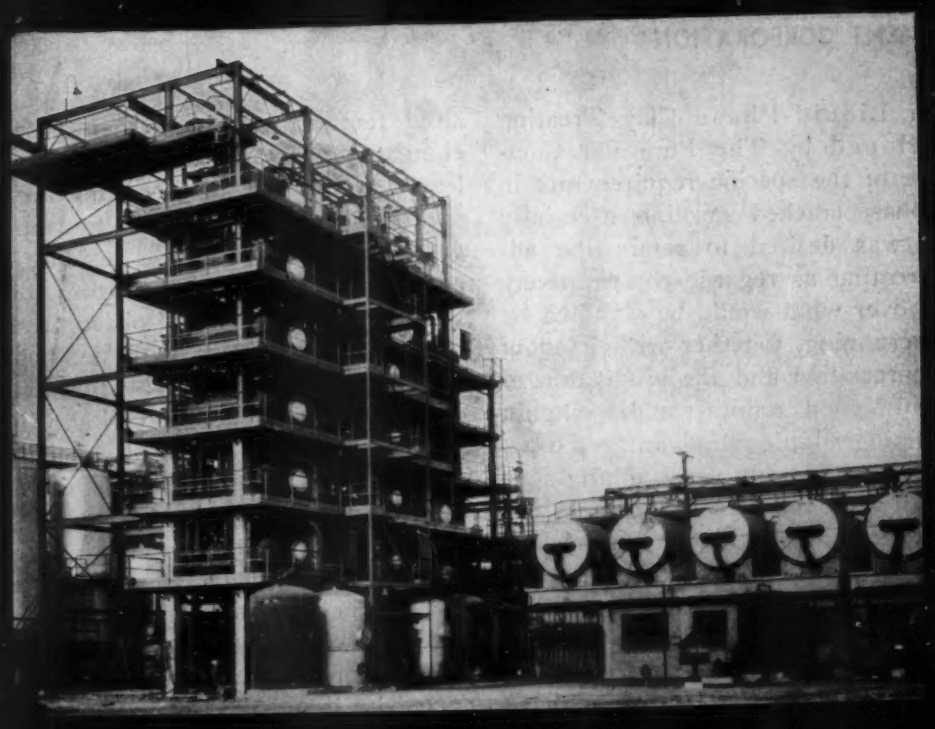
In general, oil of any quality up to Pennsylvania grade or better can be obtained from any distillate or residuum which contains any oil of this quality. The quality of the lubricating oil obtained is dependent on the degree of treatment.

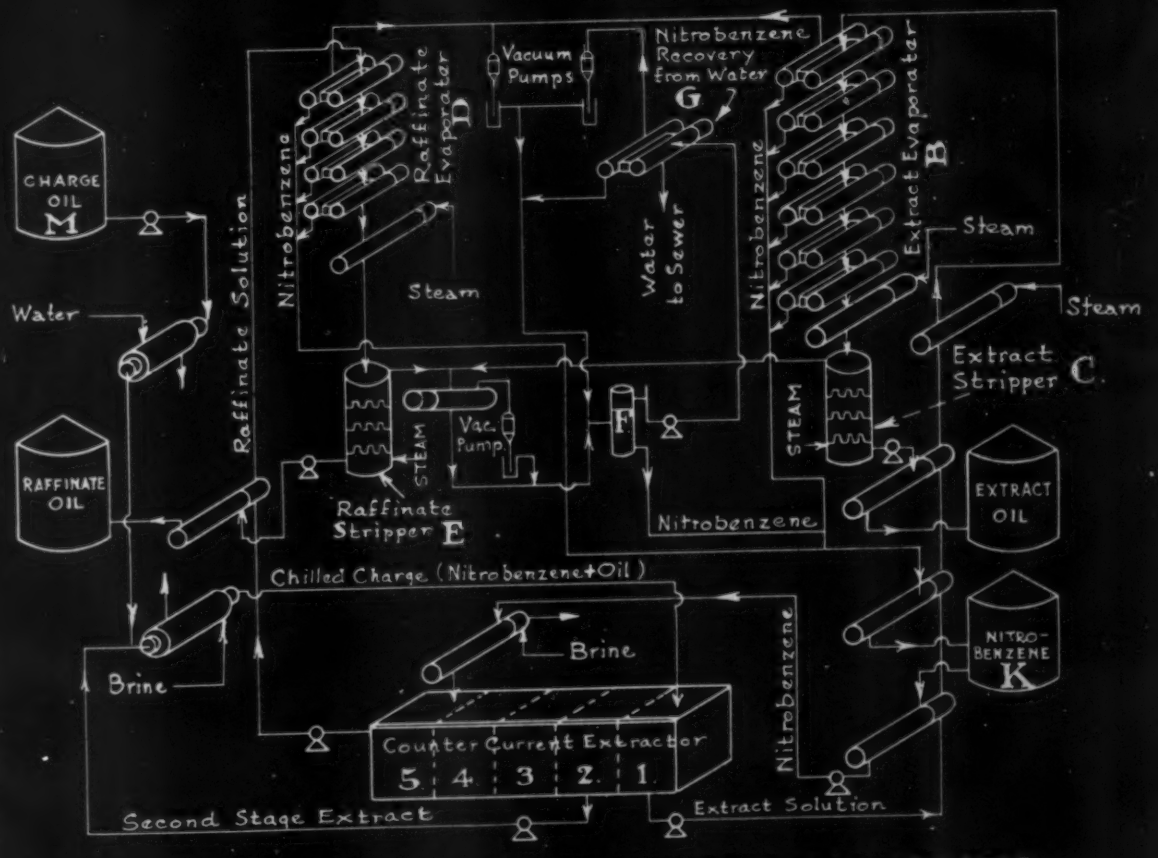
Charge stocks varying from light distillates (200 S. S. U. viscosity at 100°F.) to heavy residuums (950 S. S. U. viscosity at 210°F.) have been successfully processed at an average rate of about 2000 barrels per day in the plant at Philadelphia. The color reduction obtained ranges from 60 to over 90 percent. The yield depends on the composition of the charge stock and the quality of product desired. The quantities of nitrobenzene required vary from one-half volume per volume of charge oil upward and rarely exceed two volumes per volume of charge oil.

The costs incident to the operation are: steam for heating and for distilling solvent, water for condensing and cooling, power for pumping, mixing and refrigeration, a small amount of makeup solvent, and sufficient labor to follow the operation of a continuous, automatically controlled, low temperature, low pressure plant.

The paraffinic oil made will have a higher A. P. I. gravity, a better V. G. C. and V. I., a much lighter color and lower carbon residue than the charge stock. The extent to which these improvements are carried depends on the degree of treatment used.

Nitrobenzene treatment removes those compounds which decompose in an engine to form sludge and produce sticking rings, loss of power and generally unsatisfactory operation. Hence a nitrobenzene treated oil has remarkable stability in use, minimizes the formation of sludge in the engine and provides continuous lubrication.





FLOW DIAGRAM OF NITROBENZENE EXTRACTION PROCESS

COURTESY, ATLANTIC REFINING COMPANY

Osterstrom Clay Treating Process

TEXACO DEVELOPMENT CORPORATION

THE Osterstrom Liquid-Phase Clay Treating Process was developed by The Pure Oil Company to meet some of the specific requirements in refining of vapor-phase cracked gasoline, especially Gyro gasoline. It was desired to retain the advantages of clay treating as regards comparatively low treating losses over what would be obtained by acid treating and rerunning, together with freedom from anti-knock depreciation and the production of a finished gasoline of good stability and low gum content, while obtaining additional advantages especially in the treatment of highly cracked products.

The process finally arrived at was to pass the cracked gasoline, after condensation and separation of fixed gas, through a pipe still in which it was heated to temperatures well above its boiling point at normal pressure while maintaining it at a pressure of the order of 1000 pounds per square inch. Time of passage through the coil may be long enough to effect initial polymerization due to the heat and pressure. The oil upon leaving the furnace is passed without reduction of pressure and temperature through a chamber containing a bed of fuller's earth. On leaving this chamber the pressure is reduced and the oil is passed into an evaporator tower where it is flashed and fractionated.

The oil charged to the treater may be either an endpoint distillate or higher boiling, in view of the final fractionation obtained. Polymers are drawn from the bottom of the fractionating tower and may then be returned to the cracking still for recracking.

The treatment of vapor-phase cracked distillate calls for a process which is flexible and one which treats either freshly made distillate or distillate which has been stored for some time. The Osterstrom Process is flexible in that the temperature as well as time of contact can be varied so that it is possible to treat a distillate of any age. The heating means is independent of the cracking still, so that the treating unit can be run while the cracking unit is not in operation and vice versa.

The flow diagram shows a typical arrangement. Raw distillate to be treated is pumped at approximately 1000 pounds pressure through the radiant section of a pipe still, where it is brought to the desired temperature. It is then passed through the elongated chamber, which is charged with coarse leased and the vapors resulting are fractionated. If additional heat is required to completely separate gasoline vapors from the residue, the oil may be passed through a second heating coil as shown.

The Osterstrom liquid-phase process has been in use for several years with uniformly good results as regards gum content by the air jet method and oxygen bomb induction period. Although in most plants where it is installed, color specifications are such that the treated product is satisfactory with colors ranging from 17 to 22, the treating conditions can be so modified as to yield a finished gasoline of any desired color characteristics. In one plant a color of 27 or better has been uniformly obtained. In one plant one charge of clay was in use for approximately 4½ months. Due to the long time of contact effected by liquid phase treatment as compared with vapor phase, small treating vessels can be used so that, despite the high pressures, the initial installation cost is moderate.

Following is representative data from the operation of a commercial unit:

REPRESENTATIVE HIGH PRESSURE CLAY TREATING DATA

Gyro Distillate from Mid-Continent and Michigan Reduced Crude Oils

Operating Data—

Throughput, barrels per day	2500-4000
Pressure, pounds per sq. in	1000
Temp. tube still out, deg. F.	550- 580
Temp. clay chamber out, deg. F.	580- 630
Total charge, barrels	166,342

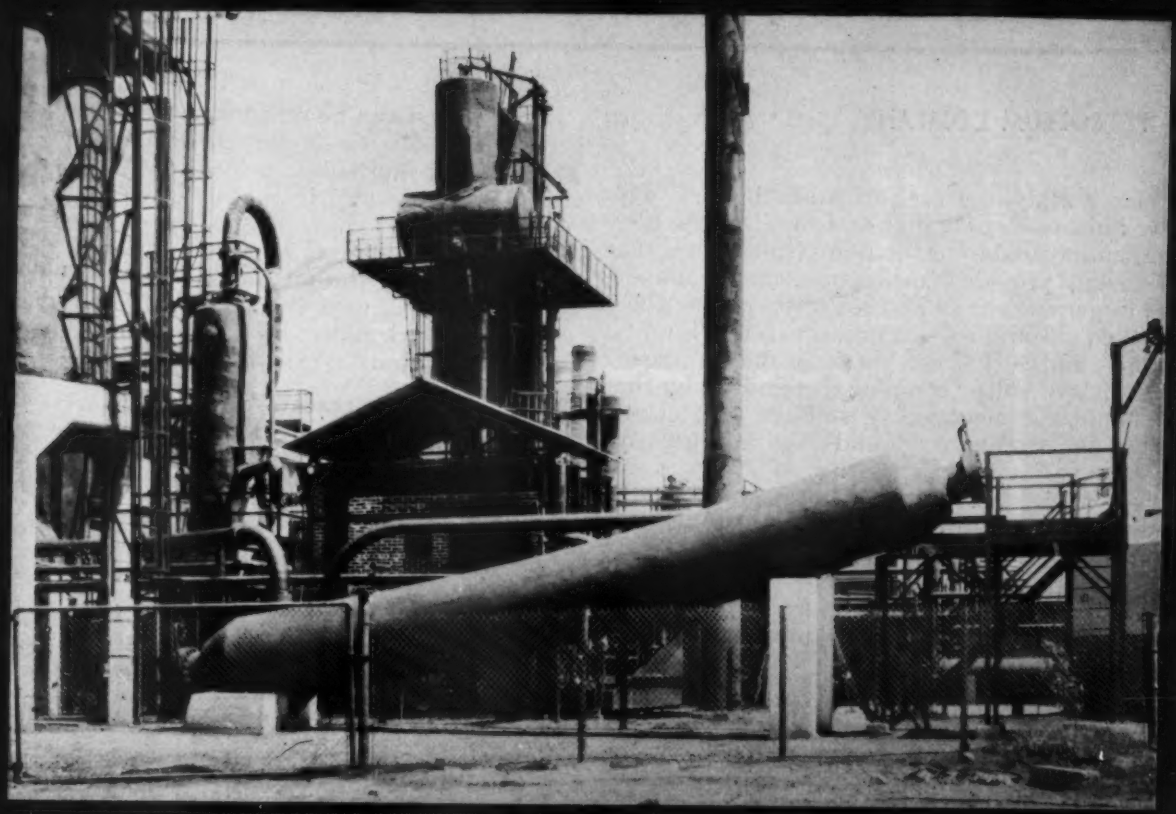
Clay Yield—71,947 barrels of finished gasoline per ton of clay.

Quality of Products—

	Grav.	I.B.P.	10%	20%	50%	90%	E.P.	% at 500° F. Color	Gum
Charging Stock	.54.4	87	136	165	263	387	435		
Finished Gyro	..54.7	90	129	166	265	393	417	16-22	2mg.
Polymers	....23.9	416	438	448				49%	

The finished Gyro showed an induction period of four hours plus.

The Osterstrom Process is licensed by Texaco Development Corporation, 135 East 42nd Street, New York.



OSTERSTROM
CLAY TREATING PROCESS

COURTESY THE GRAY PROCESSES CORPORATION

Perco Catalytic Desulfurization Process

PHILLIPS PETROLEUM COMPANY

THE Perco Catalytic Desulfurization Process developed by Phillips Petroleum Company, Bartlesville, Oklahoma, and available to the industry through the Perco Division, provides an economical means of removing, in general, 40-60 percent of the sulfur content of cracked gasolines, 60-98 percent from straight-run gasolines, and 90-100 percent from natural gasolines. The types of sulfur compounds removed by the process include the mercaptans, sulfides, and disulfudes, while the cyclic compounds are not greatly affected. The variation in quantity of cyclic compounds present in the above mentioned types of gasoline accounts for the differences in reduction of total sulfur content.

It is well recognized now that sulfur compounds in motor fuels have a very marked effect in reducing the response to tetraethyl lead. The primary purpose of catalytic desulfurization is to eliminate this effect, although there are other important benefits in many cases, such as reduction of sulfur content to specifications, improvement in odor, and improvement in stability. The improvement in lead response usually is accompanied by an improvement in the clear octane number, and the combined effect often produces a fuel to be sold in the next higher price bracket.

Catalytic Desulfurization is particularly applicable for improving the quality of naphthas to be used in the manufacture of high-octane aviation gasolines. Substantially complete desulfurization, as obtained in this process, is required for maximum response to tetraethyl lead.

The process operates at low pressure and moderate temperature. As shown in the flow sheet on the opposite page, the feed is vaporized, usually by heat exchange with the product leaving the catalyst tower, and then superheated to a temperature in the neighborhood of 750° F. The superheated vapors are passed through the catalyst bed, where organic sulfur compounds are decomposed to hydrogen sulfide. The pressure on the catalyst tower generally is maintained at 50 pounds or less, and the flow rate may range from 100 to 300 barrels of liquid feed stock per ton of catalyst per day. After cooling and condensing the product, the hydrogen sulfide is removed by whatever means is most applicable in the particular instance, such as fractionation or chemical treatment or a combination of both.

Operating cycles up to 30 days or more for a charge of catalyst are obtained on many gasolines. Catalyst cost is of the order of one cent per barrel.

For the treatment of cracked gasolines or blends containing cracked gasolines which deactivate the catalyst, auxiliary equipment is provided for catalyst regeneration.

The regeneration operation is accomplished by charging to the alternate chamber a stream of low-pressure air to burn off the carbonaceous matter, such air being in admixture with a predetermined quantity of steam superheated to give a temperature of about 750° F. for the air-steam mixture entering the reactor on regeneration.

The catalyst can be regenerated an indefinite number of times without impairment of its activity. There is substantially no loss of hydrocarbons involved in the desulfurization treatment.

The process is especially useful in the production of aviation or premium motor fuels from straight-run gasolines, where the maximum lead response is desired. Several typical examples of the effects of catalytic desulfurization on lead response are shown below for various types of gasoline.

CRACKED GASOLINE

56° API Gravity; 9-Lb. R.V.P.; 420° F. E.P.

	Undesulfurized	Desulfurized
ASTM Octane No.	65.7	69.1
+1 cc TEL	70.0	75.8
+2 cc TEL	72.4	78.0
+3 cc TEL	74.5	79.8
Total Sulfur	0.140	0.076
Mercaptan Sulfur	0.062	0.003

STRAIGHT RUN NAPHTHA

50° API Gravity; 3-Lb. RVP; 440° F. E.P.

	Undesulfurized	Desulfurized
ASTM Octane No.	45.4	49.6
+1 cc TEL	52.0	59.8
+2 cc TEL	56.6	66.8
+3 cc TEL	59.8	70.5
Total Sulfur	0.323	0.053
Mercaptan Sulfur	0.119	0.002

STRAIGHT RUN GASOLINE

58° API Gravity; 6-Lb. R.V.P.; 400° F. E.P.

	Undesulfurized	Desulfurized
ASTM Octane No.	53.8	57.8
+1 cc TEL	59.2	68.9
+2 cc TEL	63.0	74.1
+3 cc TEL	65.9	77.4
Total Sulfur	0.309	0.017
Mercaptan Sulfur	0.148	0.000

STRAIGHT RUN GASOLINE

64° API Gravity; 8-Lb. R.V.P.; 375° F. E.P.

	Undesulfurized	Desulfurized
ASTM Octane No.	58.5	60.8
+1 cc TEL	65.4	72.8
+2 cc TEL	69.4	78.5
+3 cc TEL	72.2	81.5
Total Sulfur	0.143	0.005
Mercaptan Sulfur	0.067	0.001

CRACKED-STRAIGHT RUN-POLY GASOLINE

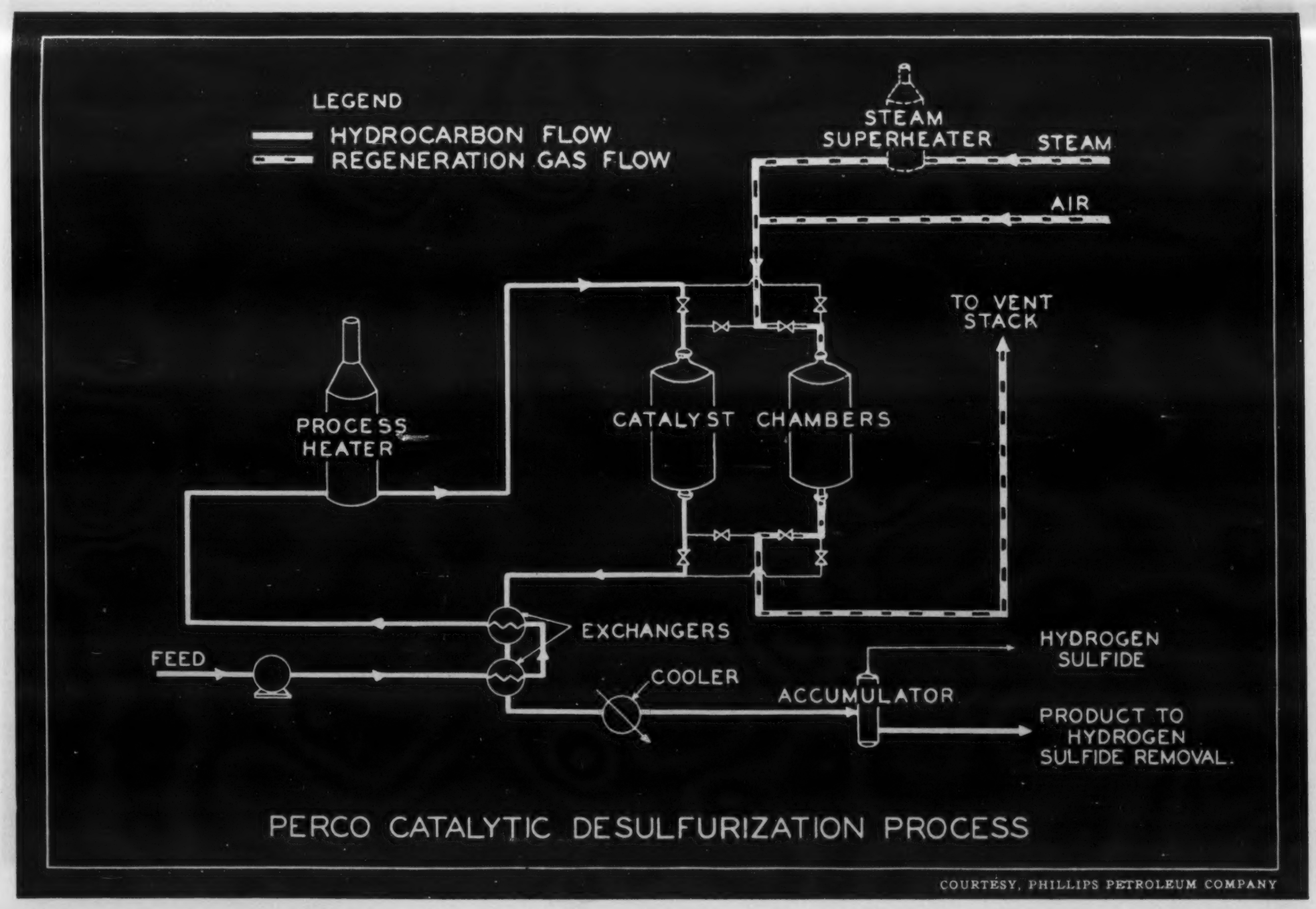
66.2° API Gravity; 9.9 R.V.P. 355° F. E.P.

	Undesulfurized	Desulfurized
ASTM Octane No.	58.6	63.9
+1 cc TEL	66.4	73.9
+3 cc TEL	73.1	79.0
Research Octane No.	61.4	67.2
+1 cc TEL	70.0	76.6
+3 cc TEL	77.9	83.7
Total Sulfur, Percent	0.088	0.024
Mercaptan Sulfur, Percent	0.023	Sweet

STRAIGHT RUN GASOLINE

68.3° API Gravity; 12.4 R.V.P. 346° F. E.P.

	Undesulfurized	Desulfurized
ASTM Octane No.	44.8	46.8
+1 cc TEL	53.9	59.6
+3 cc TEL	63.4	71.5
Total Sulfur, Percent	0.056	0.005
Mercaptan Sulfur, Percent	0.024	0.003



PERCO CATALYTIC DESULFURIZATION PROCESS

COURTESY, PHILLIPS PETROLEUM COMPANY

Perco Catalytic Reforming Process

PHILLIPS PETROLEUM COMPANY

THE Perco Catalytic Reforming Process developed by Phillips Petroleum Company, Bartlesville, Oklahoma, and licensed to the industry through its Perco Division, provides an economical means of both desulfurizing and catalytically reforming cracked and straight-run naphtha.

The normal benefits derived from Perco Catalytic Desulfurization elsewhere described in this Process Handbook, are obtained together with an appreciable increase in clear octane rating due to some additional mild cracking or reforming as evidenced by the production of fixed gases in the reactor effluent stream.

Normal process cycles are of the order of 24 hours or more with only a small deposition of carbon or other carbonaceous matter on the catalyst as compared to conventional catalytic cracking. This low carbon deposition on the catalyst permits an operation on relatively long process cycles and low-cost regeneration of the catalyst.

The process operates at low pressure, usually of the order of 50-100 psig. As shown in the accompanying flow sheets, the feed is vaporized, usually by heat exchange with the reactor effluent, and then superheated to a temperature of about 1000° F. The superheated vapors are passed through the reactor, thence to exchange with the feed and finally to product cooler and accumulator.

The regeneration of the catalyst is the same procedure as that described for Perco Catalytic Desulfurization and comprises burning off the carbonaceous deposits with a superheated air-steam mixture.

Typical Perco Catalytic Reforming results are given in the accompanying tables. Note the appreciable increase in "jump" octanes or the spread between ASTM and Research Octane, a feature of extreme interest to refiners having only thermal-type equipment. It is of further interest to note that the process is not only applicable to stocks having moderately high total sulfur but also to those stocks having relatively low sulfur.

Fixed gas losses are relatively low and yields are high, particularly in view of the C_4 and C_3 components recoverable as poly-feed.

The process may be operated either as a catalytic reforming unit or as a catalytic desulfurization unit.

ILLINOIS STRAIGHT-RUN NAPHTHA
54.7° API GRAVITY; 0.8 RVP; 240-375° F. Boiling Range

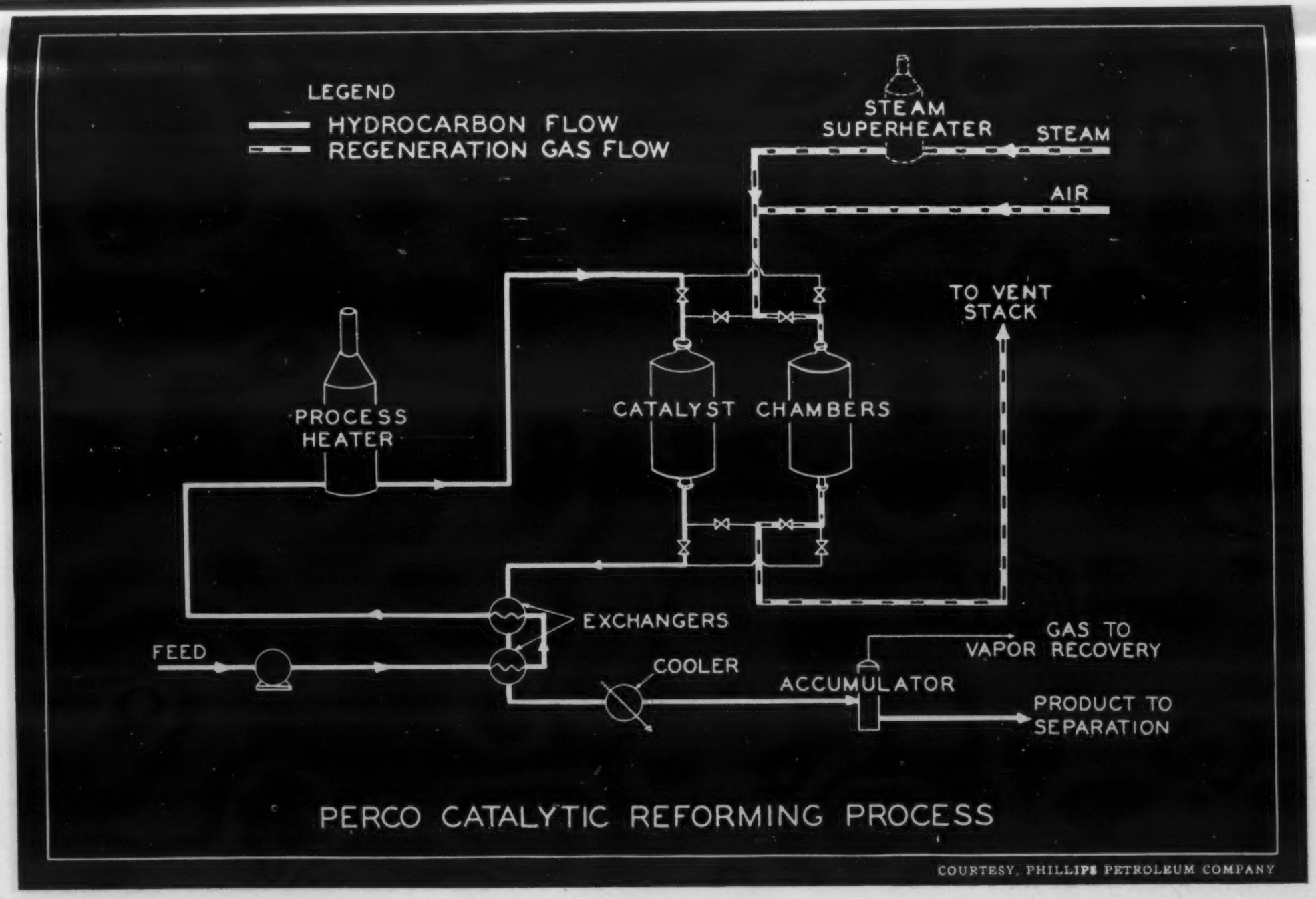
	Raw Feed	Cat. Reformed
ASTM Octane No.	36.7	45.6
+1 cc TEL	47.3	59.0
+3 cc TEL	59.4	70.8
Research Octane No.	33.8	47.8
+1 cc TEL	44.8	61.0
+3 cc TEL	58.8	72.4
Total Sulfur, Percent	0.022	0.008

PANHANDLE CRACKED-STRAIGHT-RUN GASOLINE
59.2° API Gravity; 4.8 RVP; 117-390° F. Boiling Range.

	Raw Feed	Cat. Reformed
ASTM Octane No.	57.2	62.7
+1 cc TEL	63.8	71.4
+3 cc TEL	71.6	77.7
Research Octane No.	59.5	67.1
+1 cc TEL	66.9	75.7
+3 cc TEL	74.8	83.3
Total Sulfur, Percent	0.082	0.032

EAST TEXAS DISTILLATE
66.1° API Gravity; 6 Lb. RVP; 100-410° Boiling Range

	Raw Feed	Cat. Reformed
ASTM Octane No.	47.8	70.7
+1 cc TEL	59.9	78.4
+3 cc TEL	73.0	83.3
Research Octane No.	47.0	75.7
+1 cc TEL	57.1	85.1
+3 cc TEL	72.8	90.8



PERCO CATALYTIC REFORMING PROCESS

COURTESY, PHILLIPS PETROLEUM COMPANY

Perco Copper Sweetening Process

PHILLIPS PETROLEUM COMPANY

VARIOUS methods of treating gasolines with reagents comprising compounds of copper have been suggested for many years. Phillips Petroleum Company, through licensing activities of the Perco Division, Bartlesville, Oklahoma, has made available copper sweetening processes used by over 100 refineries and gasoline plants in the United States, and units are in use in Canada, South America, Europe and Africa.

These processes take advantage of the oxidation of mercaptans by cupric chloride, and the subsequent regeneration of the cuprous chloride with oxygen as shown by the equations:

(1) 2 RSH (Mercaptan) + 2 $CuCl_2$ (Cupric Chloride) → RSSR (Disulfide) + 2 CuCl (Cuprous Chloride) + 2 HCl (Hydrochloric Acid)

(2) 2 CuCl (Cuprous Chloride) + 2HCl (Hydrochloric Acid) + 1/2 O_2 (Oxygen) → 2$CuCl_2$ (Cupric Chloride) + H_2O (Water)

(1) + (2) 2 RSH (Mercaptan) + 1/2 O_2 (Oxygen) → RSSR (Disulfide) + H_2O (Water)

As shown by the equations, one of the advantages of these processes is the theoretical absence of chemical consumption in the sweetening step. In practice it is necessary only to make up mechanical losses of the treating solution. Other operating charges are reduced, particularly labor costs, because of the simplicity of the method.

The elimination of gasoline loss, due to treating in a closed system and complete separation from the treating solution, contributes substantial savings. Probably the most important item is the improvement in quality of the copper-treated product. The product is also more responsive to gum inhibitors. The product is unusually stable when exposed to sunlight, with respect to both color and to corrosion test. Copper sweetening can form only the disulfides, since no additional inorganic sulfur is present in any form during the reaction. In the treatment the sulfur content is, of course, unchanged.

The Perco processes are applicable to any stock requiring sweetening and are especially suitable for those of a very sour nature since these are handled with only a slightly higher cost due to increased air requirements. This method of sweetening is applied in two ways; if the stock to be treated is sufficiently stable to color and gum when treated in presence of air, it is mixed with the necessary air and filtered through a bed of absorbent material impregnated with the copper reagent; other stocks are continuously treated with the copper solution in the absence of air and the copper regenerated in a separate tank. The oxidizing power of the solution is controlled to prevent side reactions with unstable hydrocarbon constituents. Stocks treated with the copper solution are given a final sodium sulfide wash to give color and gum stability, by removing minute traces of copper which may be present in the treated oil.

The simplified flow diagrams illustrate the two methods. In either process, if the gsoline to be sweetened contains hydrogen sulfide or free sulfur, these are removed by a caustic-washing step prior to the sweetening operations. If hydrogen sulfide and free sulfur are not present, this step may be omitted.

In the Perco solution process, the gasoline is continuously treated by contacting with the treating solution in a centrifugal pump. The mixture of copper solution and sweetened gasoline passes to a separating tank, from which the used copper solution is continuously withdrawn to the regenerating tank where it is regenerated with a controlled amount of air to give the desired oxidation potential. The maximum efficiency of the air is obtained by diffusers, as illustrated. The regenerated copper solution overflows to a solution-storage tank and is then recycled. The sweetened gasoline is continuously withdrawn from the top of the separating tank and is given the final stabilizing wash, settled, and passed to finished storage. In the solid process, the feed, free of hydrogen sulfide, is mixed with a predetermined quantity or air, which is dissolved in the gasoline, filtered through the reagent and passed to finished storage.

In the Perco solution process all equipment in contact with the copper solution is constructed of corrosion-resisting material, as the solution is corrosive to iron or steel. In the solid process ordinary materials are used throughout.

Due to compactness of equipment and relatively small size for a given throughput, the installation costs are low.

The following example of liquid treatment of a cracked distillate having a 170-400° F. boiling range and 53° API gravity is typical of results obtained in comparison to doctor treating.

	Sour	Doctor Treated	Perco Treated
Color, Saybolt	+2	−5	+2
Octane Number:			
Without Lead	62.9	61.9	62.9
+1 cc TEL	68.5	67.8	68.5
+2 cc TEL	71.7	71.0	71.7
Induction Period, Min.:			
Without Inhibitor	130	130	180
With 0.002 Percent Inhibitor	320	190	415
With 0.004 Percent Inhibitor	485	305	640
ASTM Gum	2.6	0.4	0.0

PERCO COPPER SWEETENING PROCESS
SOLUTION PROCESS
SOLID PROCESS
CAUSTIC CONTACTOR
CAUSTIC SETTLING TANK
CHARGE SOUR GASOLINE
COPPER CONTACTOR
SEPARATING TANK
AERATOR TANK
FLOW TANK
AIR
SULFIDE CONTACTOR
SULFIDE SETTLING TANK
SWEET GASOLINE
CAUSTIC CONTACTOR
CAUSTIC SETTLING TANK
CHARGE SOUR GASOLINE
AIR STRAINER
DUAL PROPORTIONING RECORDING FLOW CONTROL
AIR MIXER
TREATING TOWER
SWEET GASOLINE
COURTESY, PHILLIPS PETROLEUM COMPANY

Perco Cycloversion Process

PHILLIPS PETROLEUM COMPANY

CYCLOVERSION or catalytic cracking developed by Phillips Petroleum Company, Bartlesville, Oklahoma, is available for license to the petroleum refining industry through the Perco Division.

This process is schematically similar to the Perco Catalytic Desulfurization and Catalytic Reforming Process elsewhere described in this Process Handbook, and as a consequence Cycloversion may be used to accomplish desulfurization, catalytic reforming and catalytic cracking.

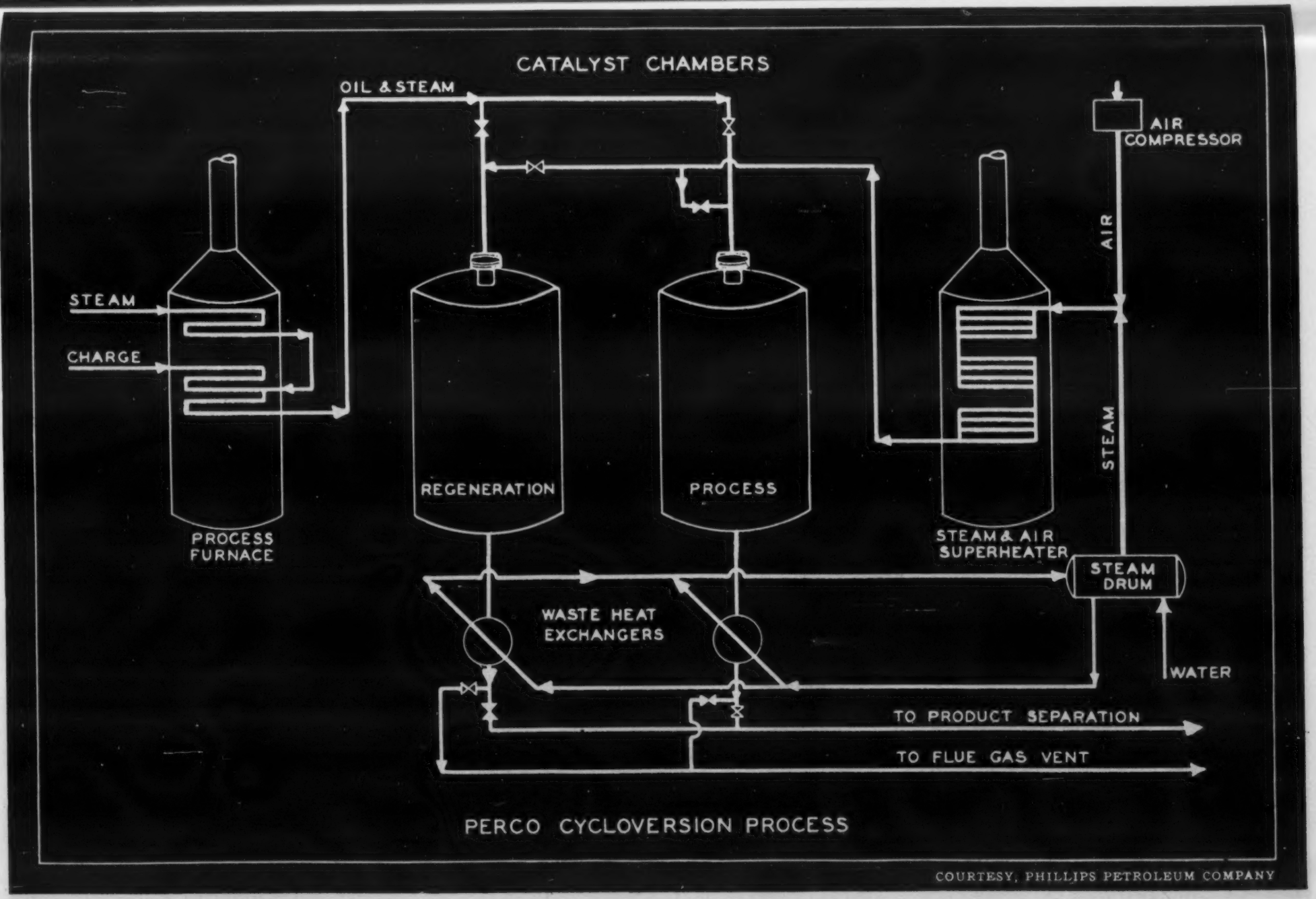
The operation of the Cycloversion Process when catalytically cracking gasoil, is illustrated in the accompanying diagram. The fresh or recycle feed is charged to a heater where it is preheated to about 980-1000° F. A small amount of steam diluent is added to the feed in the convection bank of the furnace to reduce carbon deposition on the catalyst, improve the octane rating and assist in vaporizing the charge stock. The superheated oil-steam mixture passes thence to the catalyst chamber at about 85 psig where catalytic cracking is obtained in process cycles of periods of 2-10 hours. Waste-heat exchangers are provided at the catalyst-chamber outlet to recover a large amount of heat in steam production. From the waste-heat exchangers the product passes to the heat exchange and condensing equipment and onto the oil-water-vapor separator where the condensed water is withdrawn, the vapors go to the recovery plant and the liquid hydrocarbon condensate to fractionation.

Simultaneous with the process cycle an alternate reactor chamber is undergoing regeneration. Steam from the waste-heat exchangers is superheated to about 850° F. admixed with air and charged to the chamber on regeneration. The regeneration gases are passed to waste-heat exchangers and finally vented to the atmosphere through a suitable vent stack.

The Cycloversion Process embodies a fixed-bed principle of catalytic cracking and a rugged natural baxuite catalyst capable of regeneration an indefinite number of times and maintains practically 100 percent cracking efficiency. Features of the Cycloversion Process are its low-cost operation together with high yields of superior-quality motor fuels. It is unique in that its flexibility lends it to catalytic reforming of motor-fuel-boiling-range naphthas as well as desulfurizing both cracked and straight-run products.

	Undesulfurized	Desulfurized
Feed Stock:		
Gravity, API°	36.5	36.6
10 Percent	472	510
90 Percent	735	717
Aniline No.	186	182
Catalytic Gasoline:		
Gravity, API°	60.3	60.3
Reid Vapor Pressure	10.0	10.3
ASTM Octane No.	77.0	76.3
+3 cc TEL	81.8	82.9
Research Octane No.	86.8	86.2
+3 cc TEL	93.1	93.1
Yields, Percent of Charge:		
Catalytic Gasoline 400 E.P.*	39.7	45.2
Propylenes, Vol. Percent	3.8	5.4
Butylenes, Vol. Percent	4.2	6.0
Catalytic Recycle 400+ Vol. Percent	49.5	37.1
Gas, F.O.E., Vol. Percent	9.0	12.9
Carbon, Wt. Percent	1.7	2.6
Conversion, Single Pass, Vol. Percent	50.5	62.9

* Includes Butanes after Catalytic Polymerization.



PERCO CYCLOVERSION PROCESS

COURTESY, PHILLIPS PETROLEUM COMPANY

Perco HF Alkylation Process

PHILLIPS PETROLEUM COMPANY

THE alkylation of an isoparaffin such as isobutane, with olefins, such as propylene, butylenes or amylenes, using hydrofluoric acid as a catalyst is a well-established process for producing high-octane-number premium aviation and automotive fuels.

The essential feature of the Perco HF Alkylation Process is the reaction of the olefins with an isoparaffin using liquid hydrofluoric acid as a catalyst. As shown in the flow sheet, liquid olefin-containing feed and isobutane usually are fed into the bottom of a mechanically-agitated reactor while liquid hydrofluoric acid catalyst is fed into the top. After intimate contacting in the reactor, the hydrocarbon and acid are separated in a settler. Acid is recycled to the contractor and the hydrocarbon is purified and separated by fractionation and other means.

The process is operated at ordinary atmospheric temperatures and at sufficient pressure to keep the hydrocarbon mixture liquid. The acid-hydrocarbon ratio most frequently used is in the neighborhood of 1:1. A large excess of isobutane is used over that required for reaction with the olefin. External isobutane-to-olefin ratios from 4 to 1 up to 12 to 1 have been used. The acid strength normally is maintained in the range of 85-95 percent hydrofluoric acid.

Several subsidiary operations are necessary to the process. The hydrofluoric acid catalyst is continuously purified by fractionation of a small portion of the circulating stream in an acid rerun unit as shown in the diagram. The hydrocarbon effluent is first treated to remove dissolved acid by fractionation in the azeotropic tower. The excess isobutane is removed by fractionation in a deisobutanizer, and the isobutane is separated from propane in a depropanizer before being recycled to the contactors. The deisobutanizer bottoms, containing all hydrocarbons heavier than isobutane, are catalytically defluorinated before being further fractionated. The fractionation steps following are dependent on the nature of the products desired from the process. In the flow sheet shown, normal butane, iso- and normal-pentane, aviation and heavy alkylate are all separated by fractionation.

Typical operating and other data are given in the accompanying tables. Certain features to be considered in designing a unit for peacetime motor and aviation fuel production would include:

1. Combination of the main azeotropic fractionation step with the deisobutanizing step, one fractionator serving both functions. A small azeo tower could be used on a bleed stream to a depropanization step. This change would result in savings in capital investment, utilities, and in maintenance.

2. Simplification of the acid-rerun equipment by use of a single column, acid-soluble oil and water being removed at the bottom of this column together, and by the use of a higher column pressure to eliminate the necessity of overhead-product pumps with attendant savings in maintenance.

3. In production of alkylate for motor-fuel blending, the fractionation of aviation alkylate from heavy alkylate could be eliminated in most cases with further savings in utilities and in capital investment for a new plant.

A very comprehensive treatise of the subject of HF Alkylation was published in a book by the same name by Phillips Petroleum Company, Perco Division, Bartlesville, Oklahoma, licensors of HF Alkylation.

TABLE 1

Typical Operation of HF Alkylation Unit

Charge to Unit:	Bbl. per Day
Propylene	89
Butylene	889
Amylenes	246
Total Olefins	1,224
Saturated Hydrocarbons*	
Total Feed	3,086
Recycle Isobutane, Bbl. per Calendar Day†	10,300
Yields:	**Bbl. per Day**
Aviation alkylate	2,070
Heavy alkylate	134
Isopentane	190
Normal Pentane	349
Propane	106
Acid-soluble oils	20
Aviation Alkylate Yield, bbl. per bbl. Olefin	1.7
Isobutane consumption, bbl. per bbl. Olefin	1.2
Operating Conditions:	
Isobutane-Olefin ratio (external)	7.5
Acid-Hydrocarbon ratio	1.0
Pressure, psi	150
Temperature, °F.	102
Contact time, minutes	15
Acid Catalyst:	
Titrable acidity, Wt. Percent	89.6
Oils plus solids, Wt. Percent	3.0
Water, Wt. Percent	1.6

* Contains 418 bbl. Isobutane.
† Total stream—80 percent purity.

TABLE 2

Typical Properties of HF Alkylate

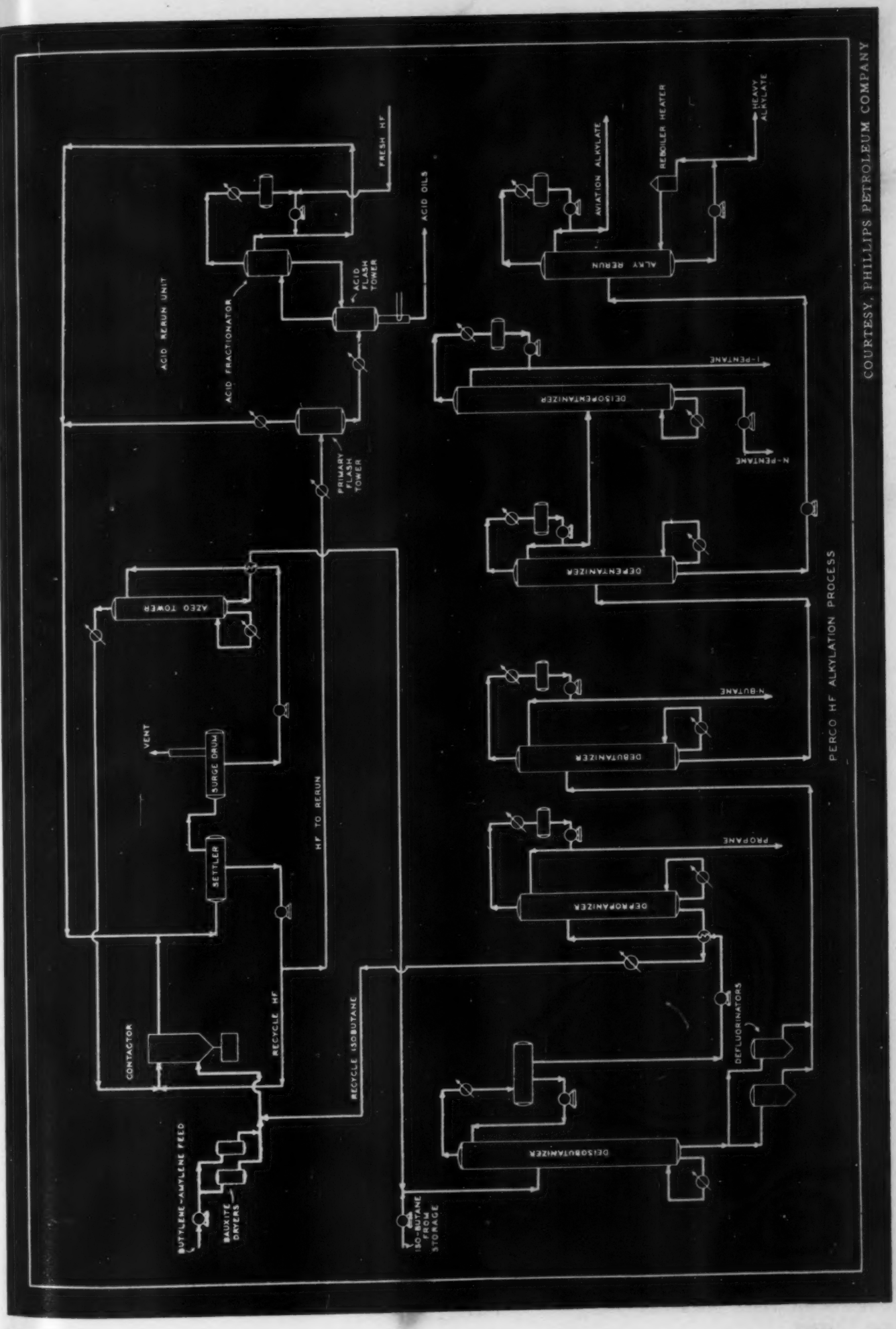
	Depentanized Aviation Alkylate	Heavy Alkylate
Gravity, °API	69.7	45.8
ASTM Distillation, °F.:		
IPB	178	416
10 Percent	200	428
50 Percent	220	448
90 Percent	239	539
End Point	327	616
Reid Vapor Pressure, lb.	2.3	....
Octane Number, ASTM	89.8	75
+1 cc TEL	100.7	
Octane Number, Research	91.6	
+ 1 cc TEL	99.7	

TABLE 3

Utility and Chemical Consumption HF Alkylation

Basis—3000 bbl. per day aviation alkylate was operation. 3400 bbl. per day motor alkylate-redesigned peacetime plant.

UTILITIES	Wartime Operation	New Design
Steam, lb. per hour	115,000	90,000
Electricity, kw	1,300	950
Water Circulation, gpm	5,300	4,600
Water, makeup, gpm	283	247
Fuel gas, cu. ft. per hr.	12,000	1,200
Chemical, lb. per month:		
Caustic Soda	9,850	9,850
Bauxite	105,000	32,000
Hydrofluoric Acid	60,000	27,000
Lime	1,500	1,500
Labor:		
Operation, man-hours per day	157	157
Maintenance, man-hour per day	105	105


PERCO HF ALKYLATION PROCESS
COURTESY, PHILLIPS PETROLEUM COMPANY
BUTYLENE-AMYLENE FEED
BAUXITE DRYERS
ISO-BUTANE FROM STORAGE
CONTACTOR
SETTLER
VENT
SURGE DRUM
AZEO TOWER
RECYCLE HF
RECYCLE ISOBUTANE
HF TO RERUN
PRIMARY FLASH TOWER
ACID RERUN UNIT
ACID FRACTIONATOR
ACID FLASH TOWER
FRESH HF
ACID OILS
DEISOBUTANIZER
DEFLUORINATORS
DEPROPANIZER
PROPANE
DEBUTANIZER
N-BUTANE
DEPENTANIZER
DEISOPENTANIZER
I-PENTANE
N-PENTANE
ALKY RERUN
AVIATION ALKYLATE
REBOILER HEATER
HEAVY ALKYLATE

Percolation Filtration Process

ATTAPULGUS CLAY COMPANY

THE percolation filtration process for finish-refining of petroleum fractions is used to produce lubricants, distillates, waxes and specialty-oil products to the high-quality specifications. Percolation-finished oils and waxes are characterized by excellent color, odor and taste, and by their stability and demulsifying properties. In recent years, improvements in equipment and more rigid application of technical control to filter-plant operation have increased processing efficiency and decreased operating costs.

Percolation filtration consists essentially of the filtering of a partially refined petroleum fraction through a columnar bed of granular adsorbent material. The adsorbents commonly used are fuller's earth and activated bauxite. Each of these materials possesses certain inherent refining advantages, depending upon characteristics of the oils being finished, and selection of the proper adsorbent is therefore the first requisite to percolation-plant design.

The mechanics of the percolation process are such that the oil and adsorbent are brought together under conditions which approach true counter-current operation. The finished products receive thereby the most thorough degree of purification.

Percolation adsorbents are revivified for reuse by burning away of the impurities adsorbed from the oils.

Lubricating-oil stocks may be refined by this process by percolation in dilution with 50 percent or more of naphtha or by the straight percolation of the oils without such dilution. Solvent-refined oils wherein the color reduction required in percolation finishing is relatively small and which are filtered primarily for stability, demulsibility, etc., usually may be filtered without dilution to high yields of finished product. A flow plan and general considerations of a plant of this latter type, suitable for the finishing of solvent-refined oils or waxes, is shown on the opposite page through courtesy of Attapulgus Clay Company, 260 South Broad Street, Philadelphia 1, Pa.

The adsorbent used in percolation finishing of mineral oils and waxes is usually a 20/60 or 30/60 mesh. The new adsorbent is heated in the adsorbent burner to 600-700° F. prior to first use. The dried or "tempered" adsorbent then is charged to the filter which may vary in capacity from 10 to 50 tons. The filter then is filled with oil and after a short soaking period the percolation proper is started. The first oil off is usually lighter colored, the stream gradually darkening until the refining power of the adsorbent may be considered to be exhausted. The filter then is drained, washed with naphtha, steamed to recover the naphtha and to condition the adsorbent for revivification. At the completion of this part of the cycle, the adsorbent is drawn from the filter and revivified in the burner at a maximum temperature of 1100° F.

The decolorizing and refining efficiency of granular fuller's earth is gradually decreased by the revivification treatment and the usual practice is to replace the plant charge at intervals. The proper point of clay discard varies with the type of oil stock being processed and is determined by cost calculations. The number of revivifications economically obtained may vary from 8 to 18 with average operating conditions and equipment.

The revivification characteristics of activated bauxite differ in several respects from fuller's earth, the most significant difference being its greater resistance to loss in efficiency caused by the revivification operation. This smaller efficiency decline obviates the necessity for periodic discard and it becomes possible to operate a filter plant under stabilized adsorbent efficiency conditions, the bolstering effect of the makeup material necessary to compensate for unavoidable handling losses just balancing the small decline in efficiency caused by revivification. The equilibrium level thus established will vary with the type of oil being treated and type of equipment used. It usually will fall within the range of 60-80 percent of the efficiency of new bauxite.

The advantages of fuller's earth are particularly evidenced in the manufacture of white medicinal oils and in the treatment of lubricating stocks where the desired color reduction is particularly high. Activated bauxite, on the other hand, displays advantageous qualities for the refining of petrolatums and waxes to light colors and to odor and taste-free specifications. The greater resistance of bauxite to efficiency degradation frequently will recommend its adoption from economy considerations. The refining ability of bauxite is useful especially in the treatment of Pennsylvania residual stock and in the finishing of special lubricating oils to high-quality demulsibility requirements.

Owing to the variable properties of oils from different crude sources and varying pre-treatments, the selection of the proper adsorbent for a given decloorizing problem should be the aim of a detailed investigation and study of the oils to be treated.

Regardless of the adsorbent employed, the percolation-filtration process possesses certain inherent merits which should be borne in mind in considering the installation of decolorizing facilities.

It is essentially a low-cost process, the greater part of the manufacturing cost comprising expense other than adsorbent cost. For this reason improvements in operating procedures are reflected in reduced decolorization costs to a much greater degree than other processes in which the adsorbent cost constitutes the major item of expense.

It is essentially a counter-current process in contrast to other decolorization methods which frequently represent the least desirable application conceivable from the standpoint of counter-current operation.

It is flexible, permitting simultaneous processing of several products in the filter house as well as production of several "specialty cuts" from the same filter.

It is an adsorption process operating at low temperatures and, as a result, yields finished oil of generaly higher quality.

Ultimate yields, that is, yield of decolorized oil in percent of stock charged, are generally high.

A recent development in percolation filter plant design has been the employment of an open plan of construction in place of the conventional walled-in filter house. This has materially decreased installation costs and has also eliminated a number of safety hazards long inherent to the older design.

PERCOLATION FILTRATION PROCESS
FLOW DIAGRAM
REVIVIFIED ADSORBENT
STEAM
CHARGE HEATERS
WASH NAPHTHA
FILTERS
SPENT ADSORBENT - TO BURNER
FINISHED OILS TO STORAGE
UNFINISHED OILS
STEAMINGS
CONDENSER
DECANTER
WATER
DIRTY NAP.
DIRTY NAP.
CLEAN NAPHTHA
WASH NAPHTHA PUMP
TO DIST. UNIT
FROM DIST. UNIT.
PLANT PLAN
ADSORBENT BURNER
SPENT ADSORBENT BIN
HOT ELEVATOR
SURGE BIN
BELT
SPENT ADSORBENT ELEVATOR
PLANT ELEVATION
SURGE BIN
SPENT ADSORBENT
BURNER
BELT
BELT
COURTESY, ATTAPULGUS CLAY COMPANY

Petreco Electrical Desalting Process

PETROLEUM RECTIFYING COMPANY

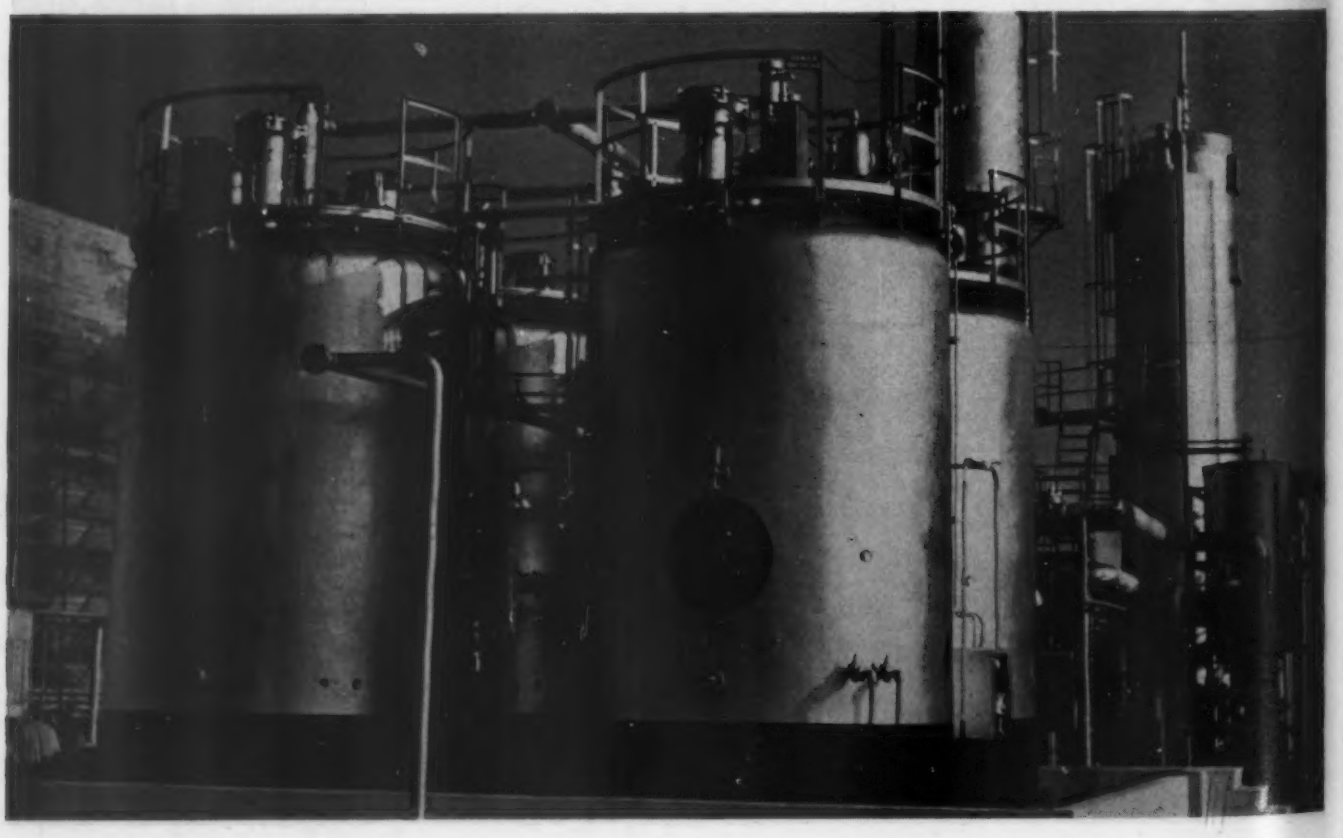
THE Petreco Electrical Desalting Process was developed by Petroleum Rectifying Company to provide an efficient and satisfactory method for removing salt and other solid materials from crude charging stock. The process is carried on by dispersing a quantity of fresh water into the salt-bearing oil stream so as to form a continuous emulsion. The emulsion so formed then is subjected to the action of a high-intensity electric field to coalesce the dispersed water into masses containing the detrimental material. The process is continuous and can be tied into the refinery stream so that the oil is purified as it goes to the stills.

The volume of fresh water required ranges from 2 to 15 percent of the volume of oil being desalted—such volume being determined by the nature of the crude oil and the concentration of salts present therein. The water is emulsified into the oil by passing the mixture through a pressure-reducing valve mounted outside and adjacent to the desalter. The pressure drop across this valve varies according to the type of oil being processed—ranging from 3 to 50 pounds per square inch. The operating temperature for the process varies from 150 to 200° F. Since the process operates by virtue of high-tension electrostatic forces, the electric power consumption is low, the average demand being 2 kw per unit, with a maximum of 10 kw. After subjection to the action of the electric field, the oil and water separate into two layers and are drawn from the desalter in two continuous streams.

The standard Petreco desalter is a cylindrical pressure tank 10 feet in diameter by 12 feet on the straight side. The tanks are set up vertically, and are about 17 to 23 feet overall in height. Specially designed high-capacity units with throughput capacities up to 50,000 barrels per day are available. Any number of the standard units can be installed as a battery, so that there is no limit to the capacity of the installation. On such multiple-unit batteries, one or two emulsifying valves handle the entire stream. Multiple units are manifolded together for automatic control of the water level. Electric-power control is maintained by individual switch gear operating at either 220 or 440 volts.

The Petreco process is in use throughout the United States, and in Canada and South America. Some of the oils desalted contain as high as 1100 pounds of salt per thousand barrels of oil; at the same time many refiners desalt oil bearing as low as 10 to 15 pounds per thousand barrels. Reduction of salts varies from 85 to 99 percent, with an accompanying reduction in all corrosive substances except sulful. It also removes effectively a large portion of suspended insoluble matter such as sand, volcanic ash, etc.

The Petreco Desalting Process is engineered, serviced and licensed by Petroleum Rectifying Company of California, Los Angeles, California.



PETRECO ELECTRICAL DESALTING PROCESS
COURTESY, PETROLEUM RECTIFYING COMPANY
TO STILLS
LEVEL CONTROL
AIR LINE
AIR OPERATED VALVE
CRUDE PUMP
STEAM TO PUMP
METER
WATER SUPPLY
SURGE DRUM
EMULSIFYING VALVE
HEAT EXCHANGER
BACK PRESSURE VALVE
CLEAN OIL
CRUDE TO TREATERS (150°-200°)
DE-SALTER
DE-SALTER
SALT WATER DISCHARGE

Phenol Extraction Process

THE M. W. KELLOGG COMPANY

PHENOL extraction is a liquid-to-liquid extraction process for improving the viscosity index and oxidation resistance, and for reducing the carbon and sludge-forming tendencies of lubricating oils made from crude petroleum.

The process is employed successfully either before or after dewaxing on both distillate and residual oils from all types of crudes. The selective solvent employed—phenol—gives high yields of desired products and has exceptional stability. The solvent is continuously re-used with losses approximating 0.01 percent of the total solvent circulated per day in well-operated plants. The high solvent power of phenol necessitates only comparatively low ratios of solvent to oil, a feature which is important in securing low operating costs.

A total of 10 phenol extraction plants now operating in the United States and Canada have a combined oil-charge capacity of about 37,000 barrels per day. Six additional units now being installed in the United States will have a combined oil charge of about 23,000 barrels per day.

Modern phenol extraction plants are continuous throughout and employ a very efficient vertical countercurrent extraction tower, the design of which permits usually high oil-charge rates per square foot of cross section. Heat requirements are kept to a minimum by utilizing heat from the condensing phenol vapors to vaporize solvent going to the extract-recovery system.

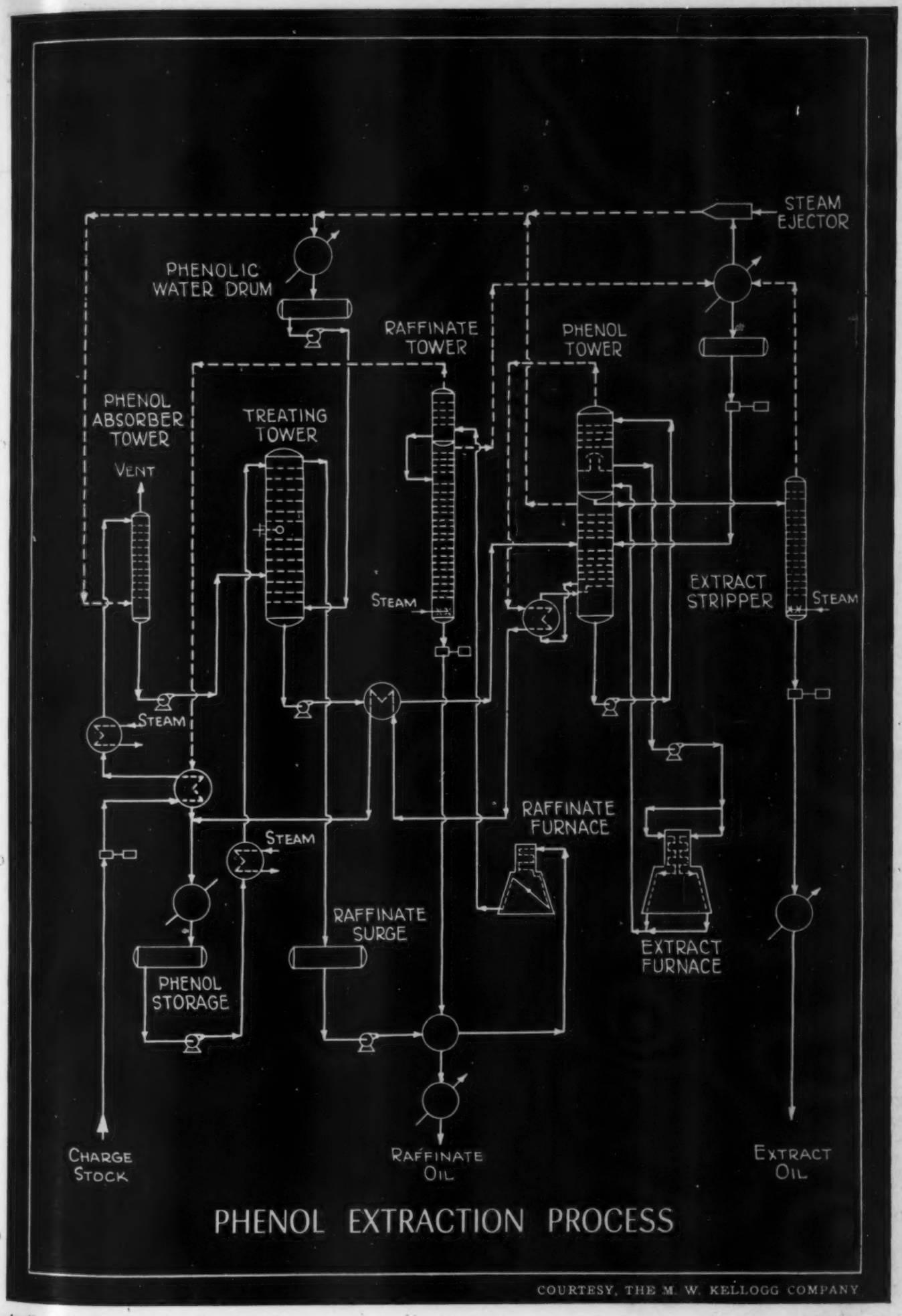
A simplified flow diagram, outlining the complete treating and solvent recovery systems, is shown on the opposite page. Raw stock and dry phenol are fed into the treating tower. Prior to entering the treating tower, the feed stock flows through the phenol absorber tower to remove the remaining phenol from process water being discharged from the plant.

Raffinate phase is withdrawn from the top of the treating tower to the raffinate storage drum from which it is fed through a heat exchanger, exchanging heat with the treated raffinate oil, thence through a coil in the raffinate furnace and is flashed into the evaporator section of the raffinate tower at substantially atmospheric pressure. Phenol vapor recovered in this section flows to the phenol condenser and enters the phenol storage tank.

The raffinate oil flows to the stripper section of the raffinate tower where it is stripped free of solvent with steam and is withdrawn from the bottom to storage. Steam and solvent vapors flow from the top of the stripping section to a surface condenser and steam jet, the purpose of which is to maintain a suitable vacuum for the stripping operation. The jet discharges sufficient stripping vapors to the phenolic water drum to maintain a constant level, bypassing the remaining vapors to the phenol absorber tower for recovery of phenol, as described above. The extract phase is withdrawn from the bottom of the treating tower and is fed through heat exchangers, in which heat is recovered from phenol vapors, and enters the drying section of the phenol tower in which all water is removed as constant-boiling phenolic water vapor. The phenolic water vapor leaving the top of this section is condensed and enters the phenolic-water drum from which is drawn reflux for the drying section, as well as the water required for modification of solvent at the bottom of the treating tower. Dry extract solution is pumped from the bottom of the drying section of the phenol tower to the top of the evaporator section of the phenol tower where a portion of the solvent is flashed off. Phenol vapor reovered in this section flows through exchangers into the phenol-storage drum. The remaining extract solution drops to a pan in the evaporator section of the phenol tower and is pumped through a coil in the extract furnace returning to a point below the pan in the evaporator section of the phenol tower. Extract oil containing a small amount of dissolved phenol flows from the bottom of the evaporator section of the phenol tower to the extract stripper where the last traces of solvent are removed by a stream-stripping operation, similar to that used for recovery of phenol from raffinate. Stripper extract is withdrawn from the bottom of this tower to storage.

Treating results obtainable with phenol extraction may be gauged from the table below summarizing yields, treating conditions and product qualities for several different stocks.

	Deasphalted East Texas Short Residue	East Texas Neutral	West Edmonds SAE 40 Stock	Deasphalted Oklahoma City Residue
Solvent Quantity, Vol. Percent	300	128	213	227
Treating Temperature, °F.	245	168	201	200
Raffinate Yield, Vol. Percent	64.5	74.1	88.2	82.2
Inspections Untreated Oil:				
°API Gravity	23.0	26.2	28.3	23.8
SSU Vis. at 210 °F.	159.0	44.5	77.4	156.6
Color—NPA	7—	2½ D	3—	4½
Carbon Residue, Percent	1.96	....	0.16	0.73
Flash COC, °F.	590	400	530	595
Fire COC, °F.	680	455	595	665
V. G. C.	.833	.842	.808	.828
Dewaxed (Zero Pour) V. I.	59	63	81	74
Raffinate Oil:				
°API Gravity	29.0	32.6	30.2	27.1
SSU Vis. at 210 °F	110.7	42.3	72.3	137.0
Color—NPA	5—	1½—	2—	3½—
Carbon Residue, Percent	0.26	....	0.04	0.24
Flash COC °F.	600	405	530	605
Fire COC °F.	660	455	595	675
V. G. C.	.795	.800	.796	.805
Dewaxed (Zero Pour) V. I.	94	93	93	93



PHENOL EXTRACTION PROCESS

COURTESY, THE M. W. KELLOGG COMPANY

Phthalic Anhydride From Petroleum

E. B. BADGER & SONS COMPANY

NEW and expanded uses made the need for phthalic anhydride acute during the war and improved manufacturing processes, as well as new raw material sources, became essential.

Outlined in the flow diagram is the first phthalic anhydride plant utilizing a petroleum product as raw material. This plant was designed and constructed by E. B. Badger & Sons Company for a progressive chemical company in this country. The design capacity of 3500 to 4000 tons annually gives this plant almost 5 percent of the total domestic phthalic anhydride production capacity.

The raw-material feed for this phthalic anhydride plant is ortho xylene supplied by hydroformer unit in an adjacent oil refinery. The ortho xylene is separated from the meta and para xylenes in the crude xylene cut produced by the hydroformer unit to give a raw material of high purity for the phthalic anhydride plant.

The liquid ortho xylene feed is pumped at controlled rates into steam-heated vaporizers. The vaporized xylene is injected directly into a stream of heated air, the mixture passing into salt-cooled converter units. Catalyst used in the converter tubes is based on vanadium pentoxide (V_3O_5) and is carefully prepared, since dusting and physical deterioration and susceptibility to poisoning have a strong effect on catalyst activity.

The reacted vapor mass leaving the bottom of the converters passes through tubular heat exchangers where the gases are cooled before passing into condensers. The crude phthalic anhydride crystals deposited in the condenser units are withdrawn at regular intervals. The spent gases passing from the condensers go into furnaces fitted with gas burners and are burned in stacks so as to eliminate fume nuisance.

The crystals of crude phthalic anhydride removed from the condensers are melted in steam-heated melt tanks, the molten anhydride being distilled batchwise in two distillation stills in series. The purified phthalic anhydride is removed as an overhead stream from the second distillation stage and passes to an aluminum storage tank. From this tank it is fed to a stainless-steel water-cooled surface for solidification. The final product in the form of chips is weighed on automatic scales and packed in paper bags.

The phthalic anhydride thus produced is of high quality, meeting all purity requirements, and comparable in every way with the best produced by the older naphthalene process.

PHTHALIC ANHYDRIDE PROCESS
COURTESY, E. B. BADGER & SONS COMPANY
ORTHO-XYLENE
CHARGE PUMP
COMPRESSOR
MIXER
AIR HEATER
CONVERTER
SALT COOLER
SALT TANK
SALT PUMP
VAPOR COOLER
CONDENSER BOXES
MELT TANK
FEED WELL PUMP
PRIMARY STILL
SECONDARY STILL
STORM TANK
FINISHED PRODUCT TANK
CHILLING MACHINE
PHTHALIC ANHYDRIDE
WASTE
EXHAUST
INERT GAS
STEAM
WATER

Precise Naphtha Fractionation Process

FOSTER WHEELER CORPORATION

IN THE Precise Naphtha Fractionation Process high-octane fractions are removed, at low cost, from straight-run naphtha.

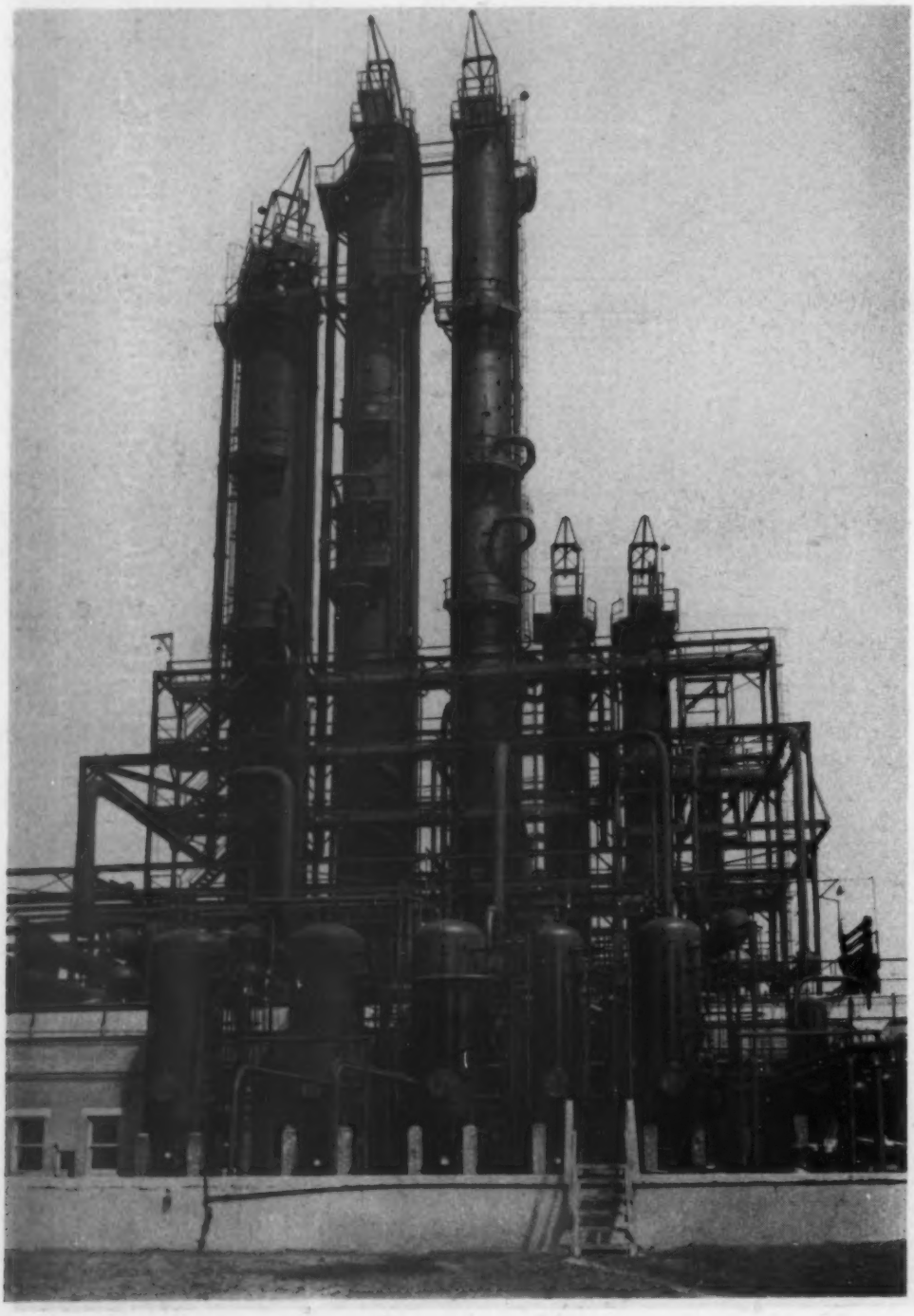
In modern refining precise fractionation of straight-run naphtha serves two purposes:

(1) Removes high-octane fractions for direct blending into motor or aviation gasolines;

(2) Concentrates all low-octane fractions for subsequent isomerization, hydroforming or reforming.

Curtailment of alkylation and isomerization activities as a result of decreased requirements for alkylate for aviation gasoline may release fractionating towers that could be adequately used for precise fractionation purposes. Similar conversions of other wartime equipment is also possible. The flow diagram on the next page shows a unit designed for the extractive distillation of toluene now being used for the precise fractionation of straight-run naphtha.

The flow diagram outlines one of several possible methods of fractionating straight-run naphtha for the purpose of separating the high-octane and low-octane fractions. The charge enters the system through a heat exchanger, is further heated in a steam heater, and enters a column where butane and lighter hydrocarbons are removed. The buttoms leaving the column flow to the next column where an iso-pentane cut is taken overhead. The buttoms from this second column flow to the third column from which the low octane fraction is removed as an overhead. The final fractionation is the sequence is in the fourth column where iso-heptane is removed as an overhead and the bottoms are pumped from the system for reforming.



PRECISE NAPHTHA FRACTIONATION PROCESS
COURTESY, FOSTER WHEELER CORPORATION
Charge
Debutanizer
Fuel Gas
C4 and lighter
Isopentane Tower
Isopentane
Dehexanizer
Low-Octane Fraction
Isoheptane Tower
Isoheptane
Reboiler
Naphtha Bottoms

Propane Deasphalting Process

E. B. BADGER & SONS COMPANY

THE deasphalting of virgin residual stocks by means of propane has become a recognized means for the production of heavy "distillate" stocks where it is necessary or desirable to hold the yield of asphalt or tar to a minimum. One adaptation of the process is outlined in the accompanying flow diagram.

The charge oil is pumped into the upper part of the extraction tower at the proper temperature, in this case approximately the temperature at which the charge is received from storage. Propane is fed into the tower at one or more points below the oil inlet, and at varying temperatures so as to give the proper temperature gradient across the tower. Additional heat to the top of the tower is supplied by steam-heated coils.

In the tower the asphalt is precipitated from the oil-propane solution, and flows by gravity to the bottom of the tower, whence it goes through a heater to the asphalt-recovery system. The asphalt phase contains a moderate amount of propane which is recovered by flashing the heated asphalt-propane mixture in the asphalt-flash drum and by stripping it with steam in the asphalt stripper. The propane-free asphalt then is pumped through a cooler to storage. If the asphalt produced is to be used as fuel and is particularly heavy, it is advisable to provide means for blending it with a light fuel oil or gasoil in order to reduce its viscosity and melting point and to avoid "freezing" in lines.

The deasphalted oil together with the bulk of the propane flows from the top of the extraction tower to the deasphalted-oil-mix evaporator. Because of the quantity of material to be handled in this particular installation the mixture is heated in vertical-tube exchangers and flashed to a special evaporator drum containing a bottom section which is also steam heated. In other installations reboiler-type evaporators may be used. From the evaporator the deasphalted oil goes to the deasphalted-oil stripper, where the last traces of propane are removed by stripping with steam. From the stripper the deasphalted oil is pumped through a cooler to storage.

The propane-steam vapors from the two strippers are sent to a spray condenser in which the water is removed. The propane vapors are compressed and combined with the dry propane from the deasphalted-oil-mix evaporator and the asphalt-mix flash drum, condensed, properly cooled and sent to the propane accumulator.

PROPANE DEASPHALTING UNIT
COURTESY, E. B. BADGER & SONS COMPANY
EXTRACTION TOWER
PROPANE HEATER
D.A. OIL MIX EVAPORATOR
D.A. OIL MIX 1 & 2 STM HEATER
D.A. OIL STRIPPER CHARGE HEATER
D.A. OIL STRIPPER
ASPHALT STRIPPER
ASPHALT MIX FIRED HEATER
ASPHALT MIX FLASH DRUM
SPRAY CONDENSER
PROPANE CONDENSER
PROPANE ACCUMULATOR
PROPANE COMPRESSOR
PROPANE CHARGE PUMP
D.A. OIL PRODUCT PUMP
ASPHALT PRODUCT PUMP
PRODUCT COOLERS
D.A. OIL
ASPHALT
CHARGE
CHARGE PUMP
STM
FUEL

Propane Deasphalting Process

THE M. W. KELLOGG COMPANY

THE propane deasphalting process is a liquid-to-liquid extraction process employing propane as a solvent for the purpose of separating asphalt, color bodies, and other undesirable constituents from reduced crudes, and for fractionating the clean oil thus produced into portions having different viscosities which may be subsequently further refined to finished lubricating oils.

The early propane deasphalting plants were of the so-called stage type, in which the reduced crude was contacted with propane in only one or two stages without appreciable counter-current action. These earlier stage-type plants made possible the separation of a considerable amount of hard asphalt from reduced crude but did not permit the production of deasphalted oils having light colors and low carbon residues suitable for subsequent refining with single selective solvents. Deasphalting plants installed in recent years have all been of an improved type with the older stage-settlers replaced by a vertical baffled tower. By employing a countercurrent flow of oil and propane within the tower and by properly controlling the temperature of the solution in the different portions of the tower, reduced crude is accurately fractionated in a series of stages into clean, light-colored deasphalted oil and high-melting point, relatively oil-free asphalt. It is probable that all future propane deasphalting plants will be of the tower type and it now appears that this process will come into general use as the primary step in manufacturing high-viscosity lubricating oils from reduced crudes.

The ability of the process to produce oil of excellent color and low carbon residue is completely independent of the asphalt content of the reduced crude. This fact, coupled with a low operating cost, makes it possible to produce lubricating oils commercially by this process from reduced crudes which have previously been considered unsuited for the purpose. Reduced crudes containing as much as 50 percent asphalt are presently being handled by this process and light-colored, low-carbon residue deasphalted oils are being produced therefrom.

Although the propane deasphalting process has been employed to date primarily for the purpose of separating reduced crude into asphalt and a deasphalted oil fraction, the process is also employed for the fractionation of the deasphalted oil into two or more portions. By a simple variation of operating conditions, the highly viscous oil portions of reduced crudes may be separated into as many different fractions as is desired and each fraction subsequently utilized for the purpose for which it is best suited. Because all separations thus made with propane are carried out at relatively low temperatures, no cracking occurs and the fractions secured are free from thermal decomposition products.

A simplified flow diagram of the propane deasphalting process is shown on the opposite page. Reduced crude is pumped from storage through a steam heater into the upper portion of the deasphalting tower. Propane is pumped from the propane-storage tank into the lower portion of the deasphalting tower from where it flows upward countercurrently to the descending asphalt phase. Deasphalted oil is withdrawn overhead and flows to a low-pressure steam propane evaporator, where a large amount of propane is flashed overhead through a condenser into the propane-storage tank. The deasphalted oil-propane solution flows from the low-pressure-steam propane evaporator to the high-pressure-steam propane evaporator where the majority of the remaining propane is flashed off, joining the stream from the low-pressure-steam propane evaporator. The oil flows from the high-pressure-steam evaporator into a steam stripper, where the remaining propane is removed. Deasphalted oil is withdrawn from the bottom of the stripper and pumped to storage.

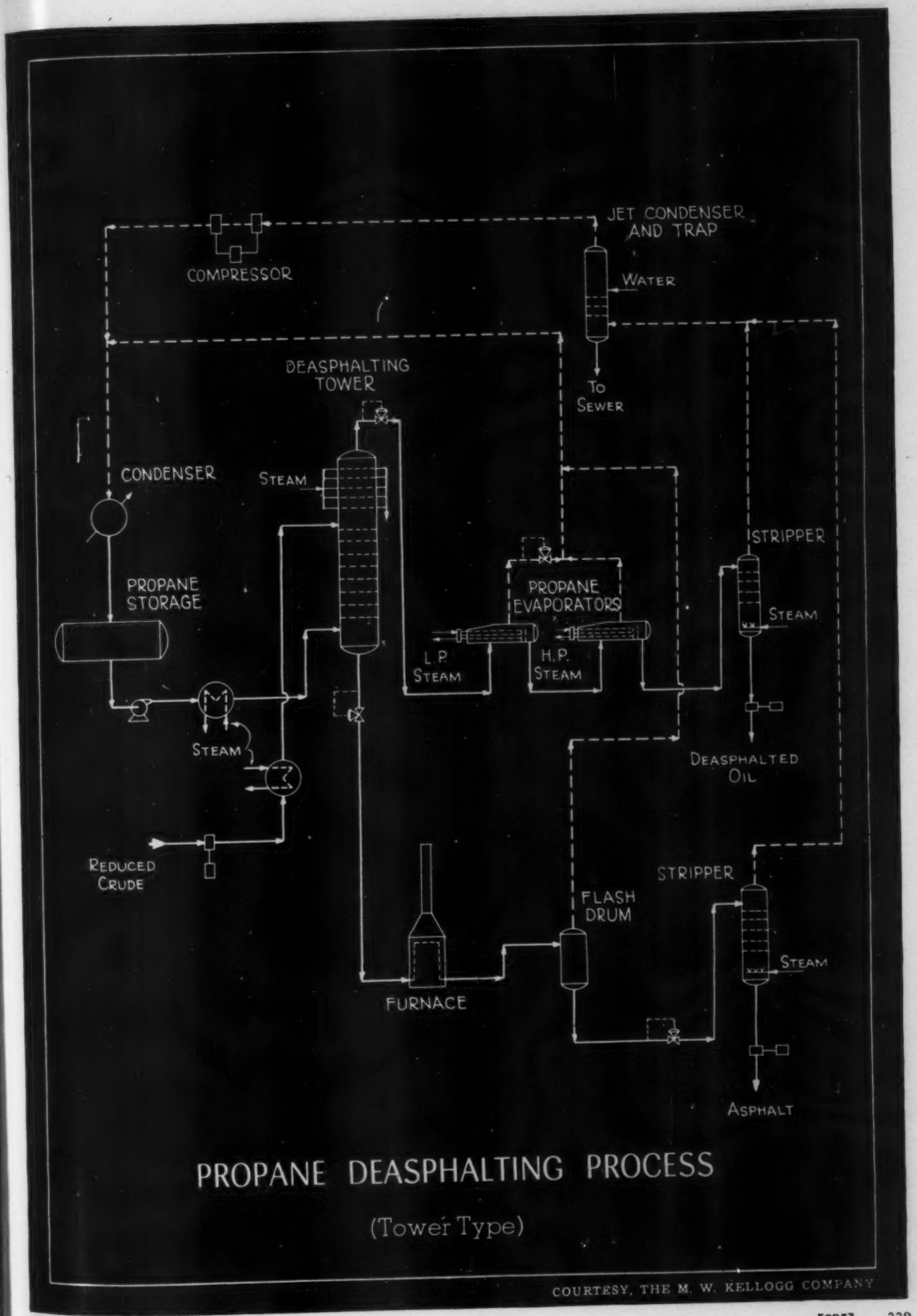
The asphalt solution is withdrawn from the bottom of the deasphalting tower and passes through a furnace and thence to a flash drum where the greater part of the propane is flashed overhead, joining the combined propane streams from the low-pressure- and high-pressure-steam propane evaporators. The asphalt with remaining small percentage of propane is withdrawn from the bottom of the flash drum into a steam stripper, where the remaining propane is removed. The asphalt product is pumped from the bottom of the stripper to storage.

The stripping steam and propane vapor from the asphalt and oil strippers passes to the jet condenser where the steam is condensed. The propane vapor goes to the compressor and thence to the propane condenser and storage tank.

Typical Yields—Tower Propane Deasphalting

	Van Zandt	California	East Texas	Heavy Mid-C.
Reduced Crude:				
°API Gravity	12.9	8.6	15.2	12.2
SSU Vis. at 210 °F	1249	7685	513	1716
Car. Res., Percent	13.9	12.1	10.9	15.2
Flash, COC °F.	585	600	575	630
Fire, COC °F.	675	690	665	695
V.G.C.	.890	.....	.882	.892
Deasphalted Oil:				
Vol. Percent Yield	58.8	32.8	63.9	54.0
°API Gravity	21.7	21.4	23.6	22.3
SSU Vis. at 210 °F.	171.9	176.4	112.9	150.8
Color, NPA	6	8—	6	5—
Car. Res., Percent	1.81	1.73	1.32	1.55
Flash, COC °F.	580	545	.....	595
Fire, COC °F.	655	650	.....	655
V.G.C.	.842	.845	.836	.840
Asphalt:				
Vol. Percent Yield	41.2	67.2	36.1	46.0
Specific Gravity	1.0583	1.0445	1.0583	1.0603
Melting Point, °F.	164	162	165	178

The M. W. Kellogg Company has been appointed licensing agent for the process for the United States and foreign countries. Licenses granted are under the patents and patent rights of Standard Oil Company (New Jersey), Union Oil Company of California, Standard Oil Company (Indiana) and The M. W. Kellogg Company.



PROPANE DEASPHALTING PROCESS

(Tower Type)

COURTESY, THE M. W. KELLOGG COMPANY

Propane Decarbonizing Process

THE M. W. KELLOGG COMPANY

PROPANE Decarbonizing is a continuous process for preparing low-carbon-forming ctalytic-cracking feed stock from reduced crude. It is the exact reverse of distillation in that the highest-boiling component is separated first. Instead of relying upon a difference in boiling points, Propane Decarbonizing effects the separation of asphalt by a difference in solubilities and at temperatures below 200° F. The asphalt separation is sharper than can be secured with vacuum distillation, and the fact that it is achieved at low temperature means that no thermal cracking occurs; thus the decarbonized oil is truly a virgin stock.

The process is distinguished by its high yields of clean catalytic-cracking stock; its ability to satisfactorily handle heavy, high-asphalt crudes which most refiners reject because of processing difficulties; and the fact that it concentrates any salt in the charging stock in the asphalt phase.

The asphalt produced as a by-product is a premium material for use in making finished asphalts for road-building, roof-surfacing, etc.

The propane lost in the process represents only about 0.1 percent of the propane circulated per day, and because of its low cost, is an unimportant factor in over-all operating cost. The total operating cost for decarbonizing catalytic-cracking feed stocks varies from four to nine cents per barrel of reduced crude, depending on the size of the unit and the cost of utilities.

A Propane Decarbonizing unit consists of a storage tank for liquid propane, charging equipment for propane and reduced crude, a countercurrent tower in which the actual asphalt separation is effected, and equipment for recovering propane.

The reduced crude is charged somewhat above the mid-point of the tower and the propane at the bottom of the tower. The majority of the asphalt is precipitated in the vicinity of the feed point. Since it is the heavier phase it flows over suitable baffles to the bottom of the tower. As the asphalt phase descends it is stripped of oil by upcoming propane with the result that only a high-melting-point, relatively oil-free, asphalt-propane solution reaches the base of the tower. This asphalt phase is continuously withdrawn, under flow control, from the bottom of the tower and proceeds to propane-recovery equipment. The oil-propane solution is further stripped of asphalt above the oil-feed point and passes from the top of the tower to its propane-recovery system.

Temperatures in the Decarbonizing Tower vary from about 120° F. to 180° F., depending on the character of the reduced crude. The heavier the reduced crude the lower the temperature at which a satisfactory separation is secured. The propane-storage tank usually is carried at a temperature corresponding to that required at the base of the tower so as to minimize the consumption of utilities.

The ratio of propane to oil by volume normally varies from a low of about three volumes of propane to one of oil, to a high of five volumes of propane to one of oil. The lower ratios usually are suitable for heavy reduced crudes containing a high percentage of asphalt, whereas the lighter reduced crudes containing a smaller quantity of asphalt require a higher propane ratio.

The majority of the propane in both the decarbonized oil and asphalt phases is recovered under high pressure and sent directly to the propane condenser without any compression being required. The small amount of propane recovered in the final strippers by the use of open steam is first sent to a jet condenser where the steam is condensed, and the propane is then compressed to condenser pressure.

Typical operating results are found in the following table.

Table of Typical Operating Results—Propane Decarbonizing

REDUCED CRUDE	West Texas	Eastern Venezuela	Mississippi	South American
Vol Percent of Crude	20.0	27.6	57.0	61.0
°API Gravity	9.1	17.0	21.3	8.6
SSU Vis. at 210 °F.	.....	195	78.5	482*
Carbon Residue, Percent	18.7	6.7	7.9	.....
Flash Point COC °F.	645	430	275	.....
Fire Point COC °F.	730	525	345	.....
Decarbonized Oil:				
Vol. Percent of Crude	8.1	22.8	49.7	30.0
Vol. Percent of Red. Crude	40.5	82.5	87.2	49.2
°API Gravity	20.4	20.6	26.0	17.5
SSU Vis. at 210 °F.	305	99.1	49.4	224
Carbon Residue, Percent	3.4	2.2	1.2	2.6
Flash Point COC °F.	625	445	.....	500
Fire Point COC °F.	710	510	.....	575
Asphalt:				
Vol. Percent of Crude	11.9	4.8	7.3	31.0
Vol. Percent of Red. Crude	59.5	17.5	12.8	50.8
Specific Gravity	1.06	1.05	1.04	1.07
Melting Point, °F.	160	156	184	187

* Saybolt Furol Viscosity

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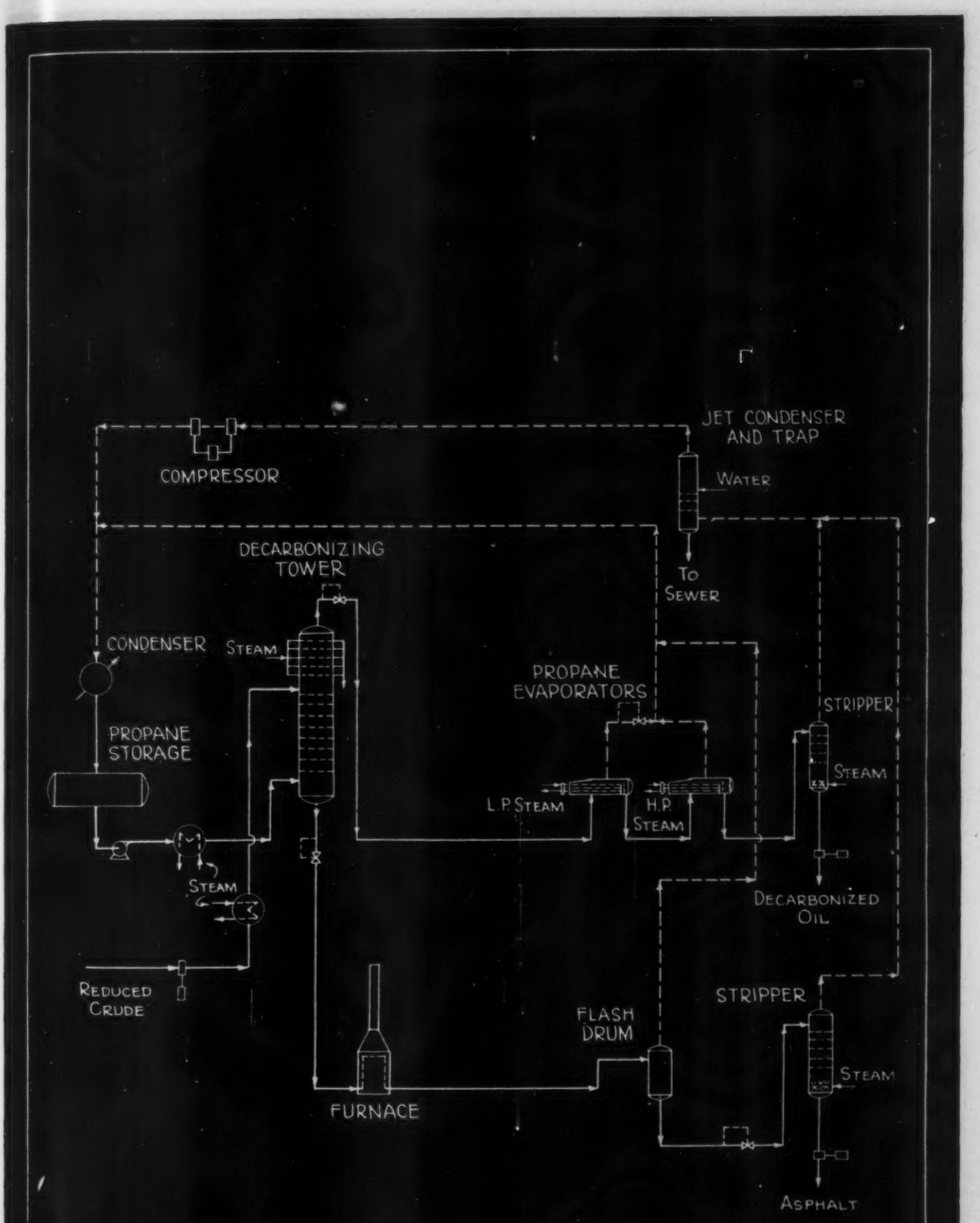

PROPANE DECARBONIZING PROCESS

(Tower Type)

COURTESY, THE M. W. KELLOGG COMPANY

Propane Dewaxing Process

THE M. W. KELLOGG COMPANY

PROPANE dewaxing is a well-known lubricating-oil process for separating wax from crude oil. The solvent, propane, is the only extraneous material used in the process since it serves both as a refrigerant and as a solvent for the oil.

Economic features of the process are its low operating cost and wide adaptability to any type of lubricating stock. High yields of dewaxed oil of zero pour test or lower and wax of low oil content are obtained.

Eight plants now operating or being built in this country have a yearly capacity of approximately 7,500,000 barrels of charging stock.

Oil and propane are charged continuously under flow control, warmed to dissolve all of the wax, and sent to a chilling vessel in which reduction of temperature to approximately —40° F. is effected by allowing propane to vaporize directly from the solution.

Wax crystallizes out at this low temperature and is separated from the oil on a continuous rotary pressure filter of most modern design.

The filter cake is washed with cold liquid propane to remove oil adhering to the wax crystals and then transferred by means of a screw conveyor and wax pump to propane-recovery equipment.

The dewaxed filtrate also is sent to a propane-recovery unit. Both end products—dewaxed oil and wax—are pumped from the strippers to storage tanks.

The low cost of propane, its double-duty as a solvent and refrigerant, and its ease of recovery are features of the process which make for high efficiency.

Propane dewaxing often is employed in combination with propane deasphalting and acid treatment in propane solution. The following table gives pertinent data on propane dewaxing of four lubricating stocks:

Propane Dewaxing Process

	Propane Deasph.-Phenol Treated 15% East Texas Reduced Crude	Phenol Treated East Texas Heavy Distillate	Propane Deasph. Phenol Treated West Edmonds SAE 70 Stock	Propane Deasph. Phenol Treated Oklahoma City SAE 40 Stock
Propane, Volume Percent	300	250	300	300
Filtering Temperature, °F.	—34	—35	—40	—41
Dewaxed Oil, Vol. Percent	61.6	67.7	85.5	81.1
Inspections Waxy Oil—				
°API Gravity	29.7	32.0	27.4	30.1
SSU Vis. at 210 °F.	93.1	57.3	132	72.7
Color—NPA	4½—	2—	3½—	2—
Carbon Residue, Percent	0.20	....	0.25	0.06
V.G.C.	.795	.790	.803	.797
Dewaxed Oil:—				
°API Gravity	27.6	28.9	26.7	28.7
SSU Vis. at 210 °F.	118.4	66.2	150.0	79.6
Color—NPA	4½—	2—	3½—	2—
Pour Point, °F.	0	0	0	—5
Carbon Residue, Percent	0.26	0.03	0.30	.05
Viscosity Index	95	92.1	91.1	92.1
V.G.C.	.804	.808	.807	.804

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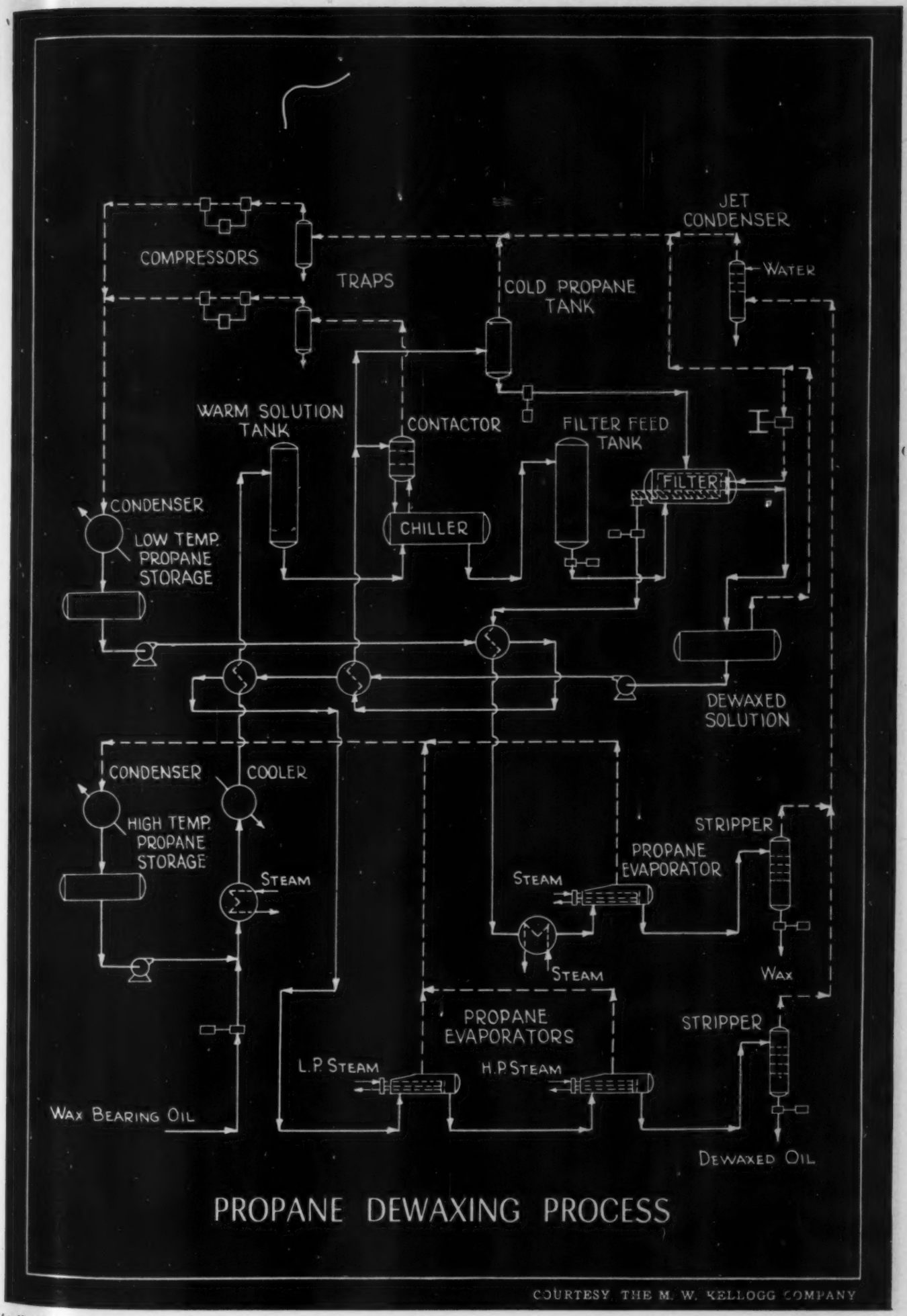


PROPANE DEWAXING PROCESS

COURTESY THE M. W. KELLOGG COMPANY

Propane Recovery Process

JONES & LAUGHLIN SUPPLY COMPANY

PREWAR absorption plants usually were designed to extract from 80 percent to 97 percent of butanes, the exact recovery sought being dependent upon the economics of the plant. Propane recovery was of secondary importance, and such propane as was recovered incidental to the selected butane recovery was marketed as such or as a component of LPG.

Since the war, the demand for both propane and LPG has, in many cases, caused propane to replace butane as the key component in the economics of plant design. The flow diagrams on the opposite page represent modern versions of two types of plants, the upper for use where butane is the key component, the lower where propane has become the key component. Both plants are flexible so that operation may be varied with market conditions to produce optimum plant returns. Stabilizer sections are omitted from both flow diagrams.

The "Moderate Propane Recovery" plant usually will recover about 30 percent of the propane, although, in the case of rich gases with a high content of heavy components, the propane recovery may go higher. The limiting factor is the still pressure, which must be kept low enough to permit stripping at the available hot-oil temperature without using uneconomical stripping steam quantities. The use of a direct-fired oil preheater as shown permits higher still pressures and, consequently, higher propane recovery.

Between the absorber and the still, a fat-oil stabilizer (or fat-oil deethanizer) is shown. Heat from normal oil-to-oil exchange is applied to the feed entering this unit and, consequently, better heat economy is secured than when exchange is applied only to the stream leaving the bottom of the fat-oil stabilizer. By controlling preheat of the lower charge stream and regulating cold oil quantities to the top and middle sections, substantially all the methane and 60 to 80 percent of the ethane can be removed from the fat-oil. This is accomplished without excessive losses of propane and heavier. Fat-oil stabilization reduces the quantity of recycle vapors from the still and stabilizers with resultant savings in equipment size and cost, while at the same time overall propane recovery is increased.

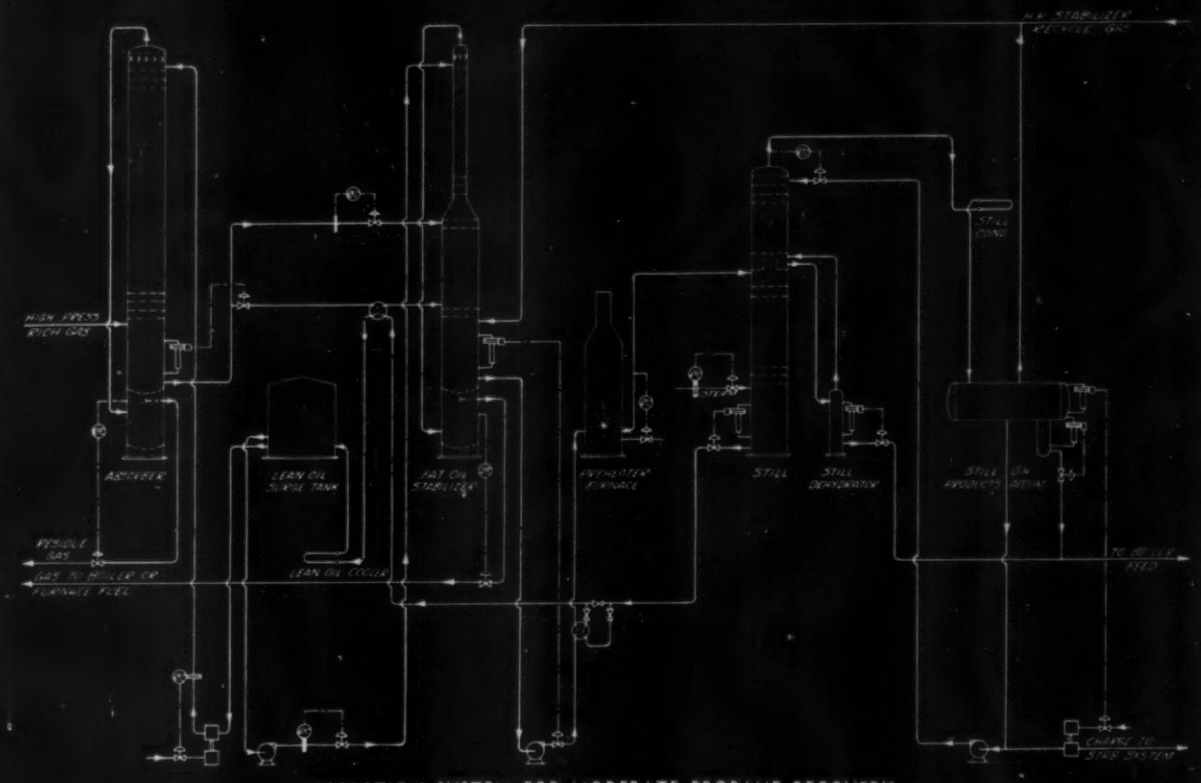
Recycle vapors that do arise are returned under their existing pressures to the bottom section of the fat-oil stabilizer and, being relatively rich in propane, they further aid in fractionating the methane and ethane from the fat-oil. In connection with this system, absorber pressure is assumed to be in the order of 200 pounds or higher.

The "High Propane Recovery" plant, indicated by the lower flow diagram, is satisfactory for propane recoveries in the order of 50 to 60 percent and may be used to retain recoveries as high as 80 percent when the charge gas is relatively rich in propane and heavier hydrocarbons. Distinguishing features of this system are the high- and low-pressure stills and the reabsorber, where the cold fat-oil serves as a large portion of the absorption medium required for the reabsorption of the heavier constituents of the relatively high quantities of recycle gases. The primary still is operated at about 225 pounds pressure in order to condense a high proportion of propane in the stabilizer-charge liquid. Since satisfactory stripping is impractical at this pressure, the primary still bottoms are fed to the secondary still, which is operated total condensing at 50 to 75 pounds pressure with the net overhead product being pumped to an intermediate point in the primary still condenser. The addition of this secondary still product aids in holding the maximum amount of propane in the liquid state. Lean-oil to the reabsorber is adjusted to secure high retention of propane. Absorber pressures of 350 pounds, or higher, are assumed.

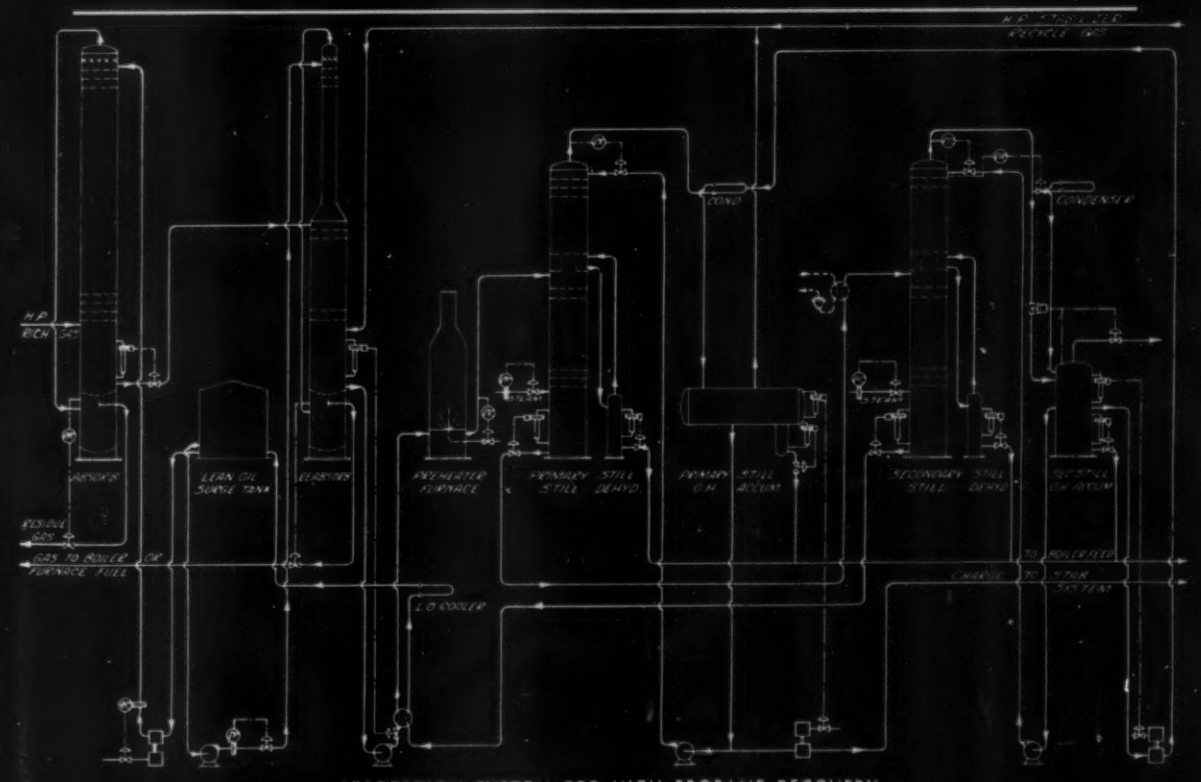
While not indicated on the flow diagram, it may be desirable in some cases to apply a small amount of heat to the base of the reabsorber to assist in the fractionation between ethane and propane and keep down the recycle gas quantities. The secondary still preheater which is dotted in on the flow diagram, may or may not be necessary, depending upon the effluent primary still bottoms temperature and the quantities of heavier gasoline components present in this previously stripped oil.

This more complicated "High Propane Recovery" plant is inherently more expensive than the first plant, but, to obtain the high pressure needed at the final condensing point, it is necessary to use either the two-still set-up or the equivalent of the primary still in recompressors. Still another possible scheme for high propane recovery is the use of refrigeration of still and deethanizer overheads. Individual plant economics will dictate the choice of method for obtaining high propane retention, but the two-still arrangement has been chosen as being the most widely applicable and the simplest in operation. First cost of the different methods just mentioned will not vary greatly.

Both the flow diagram opposite and this description were provided by Jones & Laughlin Supply Company, Tulsa, Oklahoma.



ABSORPTION SYSTEM FOR MODERATE PROPANE RECOVERY



ABSORPTION SYSTEM FOR HIGH PROPANE RECOVERY

PROPANE RECOVERY PROCESS

COURTESY, JONES & LAUGHLIN SUPPLY COMPANY

Selective Thermal Cracking Processes

THE LUMMUS COMPANY

NOW that more than a million barrels of per stream day capacity of catalytic-cracking units are drawing upon gasoils, which formerly were charged to thermal processes, the selective thermal cracking of heavy crude residues and refractory catalytic gasoils is becoming more and more important. While the solution in most plants involves adaptation of existing units to the new requirements, rather than the installation of new facilities, there still remain situations in which it is desirable to install new thermal-cracking facilities.

Selection of proper equipment often is difficult because of the availability of many types of units, as well as because of special conditions and the specific problems involved. The important factors which determine the performance and economic value of a projected thermal cracking plant may be summarized as:

Product yield and quality, initial investment, service factor, fuel and power economy, flexibility, maintenance and turnaround cost, time required for construction and time lapse between initial starting-up and acceptance.

A comparison on the basis of the above significant factors will indicate that Lummus units embody the best proven principles of the art aplied in the way best suited for the specific conditions of the refiner. The units are designed and erected under existing license agreements but they incorporate novel features and refinements of design which assure superior performance.

The yield efficiency and product quality to be expected from these cracking plants can be substantiated by commercial acceptance tests. Lummus combination cracking units are designed for the optimum segregation of furnace charge stocks and their independent selective cracking under the most suitable conditions. In some units stocks are segregated for selective cracking not only according to boiling range but also according to source or refractivity.

In order to crack selected charging stocks under optimum conditions it is necessary that the heaters be accurately designed to maintain optimum conditions of temperatures, shape of heating curve, cracking time and rate of heat transfer through the furnace. In the ordinary cracking units these limitations preclude flexibility as to charging stocks and product specifications. Lummus units meet the conditions of both flexibility and efficient selective cracking with carefully planned fractionation and recovery systems and by novel cracking-heater design. In Lummus furnaces the preheating and soaking sections are independently-fired chambers. This allows the operator a wide range of variation in cracking conditions without change in equipment and without sacrificing constancy of control for any given condition. This feature, combined with the unique method of handling combustion gases in the heating chamber makes Lummus cracking heaters outstanding in the field.

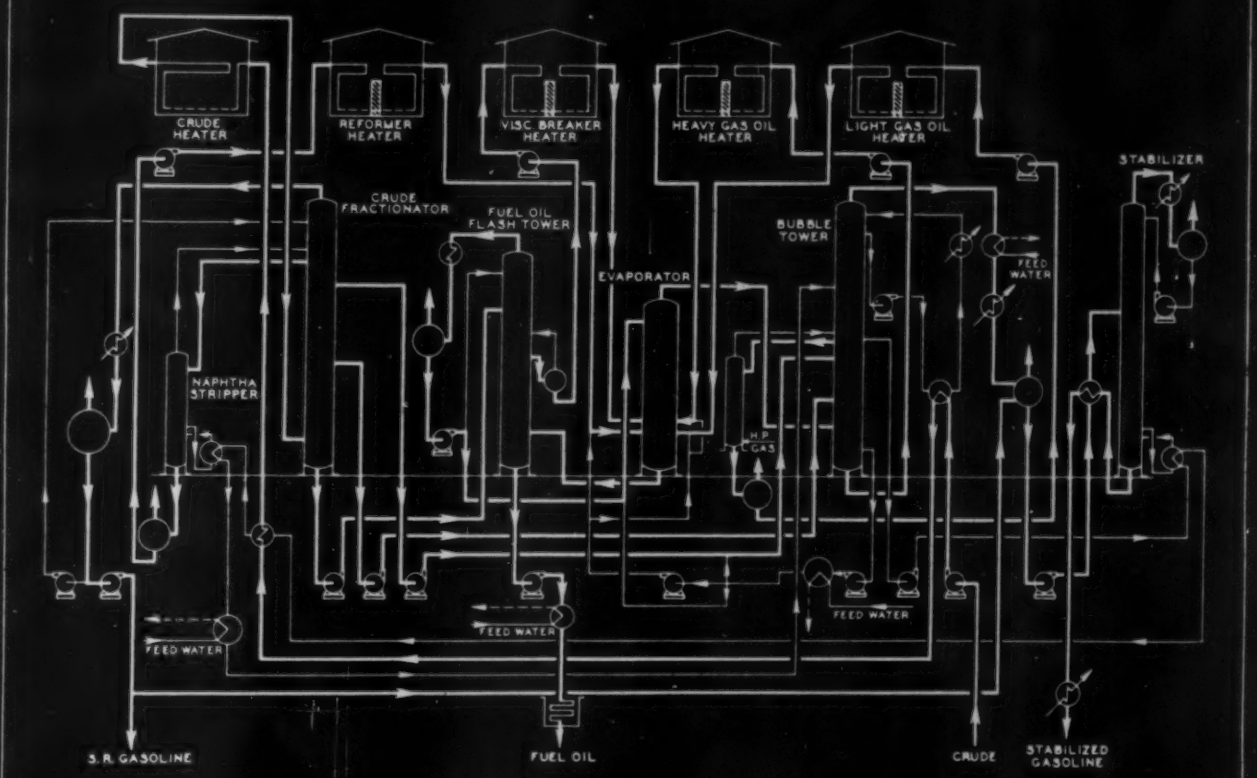
The design of the fractionating equipment of Lummus combination units reflects experience of over 40 years in the distillation field. Most combination cracking units have an excess of recoverable heat within certain temperature ranges. This heat has been utilized by generating high-pressure steam. For example, one unit generates 280,000 pounds per day of 125-pound steam. The total steam consumed by this unit is only 373,000 pounds per day. Furnace-charge pumps in this case were driven by gas engines.

Symmetry of equipment arrangement and refinements in layout pay dividends in operating convenience and rapid, inexpensive turnarounds. These features can best be appreciated by a visit to one of the units in operation.

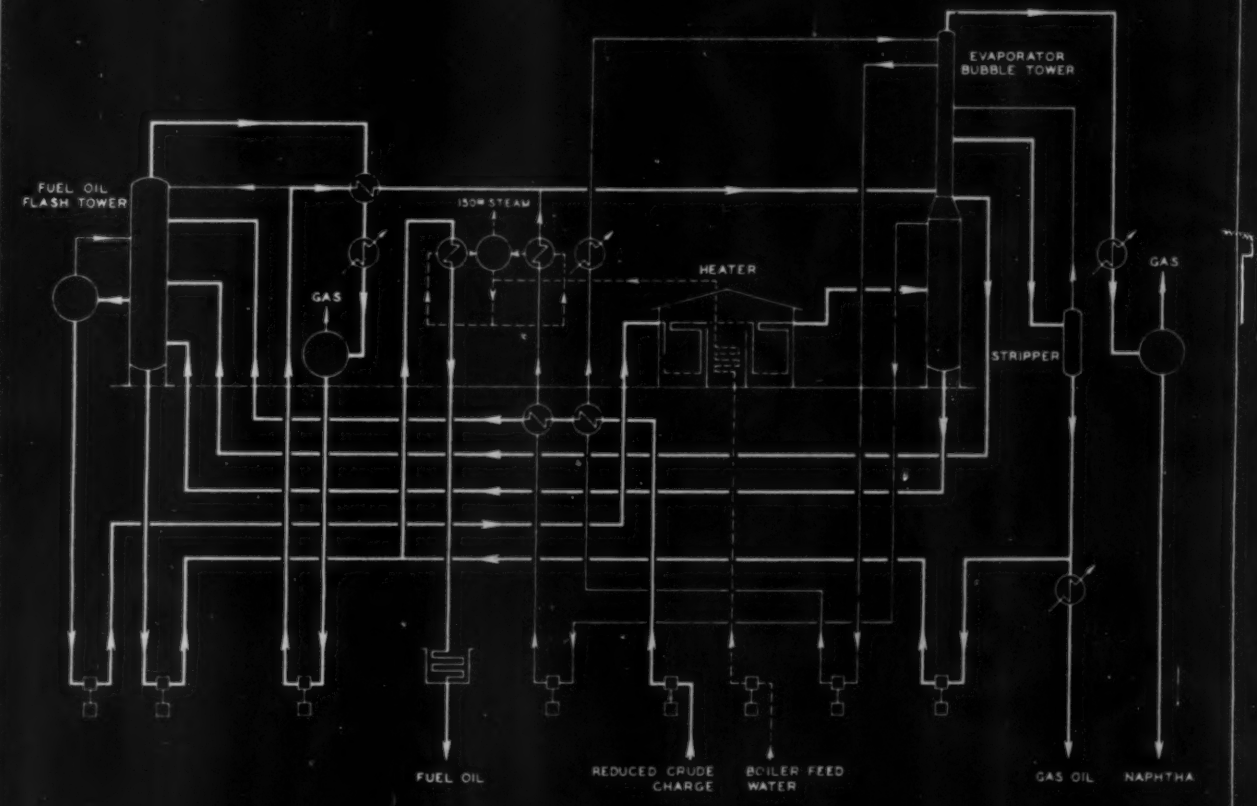
In the specialized field of reduced-crude processing, The Lummus Company offers several processes from which a selection may be made depending upon the refinery situation and marketing requirements. These processes are in general employed to increase gasoline production and reduce residual fuel oil production; they may be applied to furnish a gasoil feed for a combination or catalytic-cracking unit. Among the processes available for this purpose are Vacuum Reduction, Propane Deasphalting, Coking, and Viscosity Breaking. At the moment the urgency for reducing residual fuel oil productions is not great, as it was prior to the war because of the relatively large present demand for fuel oil and the general trend towards lower production resulting from the operation of extensive catalytic cracking capacity.

Lummus viscosity-breaking units (see flow sheet opposite) employ a number of novel features depending upon the requirements of the situation including two-coil cracking, vacuum reduction of the residue followed by cut-back with light cycle stock for the purpose of minimizing pour and viscosity, and flexibility in design which enables these units to process a wide variety of stocks efficiently.

Extensive thermal cracking facilities are now being built in one large plant by The Lummus Company, 420 Lexington Avenue, New York, which provided this discussion of the subject as well as the flow diagrams on the page opposite.



LUMMUS COMBINATION CRACKING UNIT



LUMMUS VISCOSITY BREAKER UNIT

COURTESY, THE LUMMUS COMPANY

Sharples Acid Treating Process

THE SHARPLES CORPORATION

THE Sharples Centrifugal Acid Treating Process developed by The Sharples Corporation, 2370 Westmoreland Street, Philadelphia, is used in treatment of cylinder stocks, long residuums and paraffinic and naphthenic lube distillates, non-viscous oils and light oils, such as kerosene and cracked distillate.

In this process the raw stock and acid are continuously measured in the required proportion and violently agitated by mechanical means to cause reaction; the resulting mixture is then centrifugally separated continuously, delivering sludge-free sour oil and oil-free sludge.

Important features of this process are: Thorough and rapid mixing of acid and oil; contact between oil and acid controllable as to time and temperature, which can only be accomplished by continuous operation; and high yield of oil made possible only by the use of centrifugal separation.

The specially designed proportionometer used for measuring the acid in required proportion to the oil consists of an oil meter interconnected with a combined acid pump and meter. The oil meter is a positive displacement device of the piston type which gives accurate measurement of the oil entering the system over a wide range of rates of flow, temperatures or viscosities of the oil treated.

The acid is pumped and measured into the system by a specially designed, double-acting, outside-packed piston pump.

The proportionometer will give an accurate proportional measurement of acid to oil over all rates from no flow to the maximum capacity of the combined equipment. When there is no oil flow, the acid flow is shut off entirely.

The acid and oil flow separately to a multistage mechanical mixer which provides efficient agitation and proper contact time between the acid and oil, so that the mixture leaves in a completely reacted state suitable for separation.

The mixer is equipped with ball bearings mounted independently of the mixing chamber, thus preventing contact with acid or sludge. It is driven by direct connected electric motor. The mixing chamber may be opened for examination and cleaning without removal of the mechanism used in agitation. A new improvement in the mixer consists of tubular finned heating or cooling elements which may be readily removed for cleaning or replacement.

The Sharples D-4 Centrifugal has been improved to meet the varied requirements of refinery acid treating and provides exceptionally high separating capacities. Complete oil-sludge separations are obtained at charging rates varying, with charge stock viscosity, from 400 bbls. daily in the treatment of cylinder stocks and 800-1000 bbls. daily neutrals, to 4000 bbls. daily of cracked distillate. For the oleum treatment of white oil stocks and insecticide bases, the Centrifugals are of special design and materials of construction adapted to withstand the corrosive action of oleum.

Proportionometers and mixers of specially resistant metals have also been developed for use in treating with oleum, and are suitable for metering and handling sour oil in multiple treats.

The flow chart on the opposite page shows a typical Sharples installation for the acid treatment of distillate lube stocks at an average rate of 800 bbls. daily provided with the D-4 Centrifugal for sludge separation.

The operation of the process is as follows: Raw oil is picked up from storage by the feed pump and passed continuously through the heater to the proportionometer where its measurement actuates the pumping of the required acid dosage from the acid tank. From the proportionometer, oil and acid flow in separate streams to the mixer where they are intimately mixed and the treating reaction occurs. From the mixer the completely reacted mixture passes to the Centrifugal where the oil and sludge are separated and flow separately by gravity to the sour oil and sludge tanks.

By the use of the Sharples Centrifugal Acid Treating Process the following advantages over the conventional gravity treating can be obtained.

Loss of oil to sludge is reduced by as much as 25-40 percent.

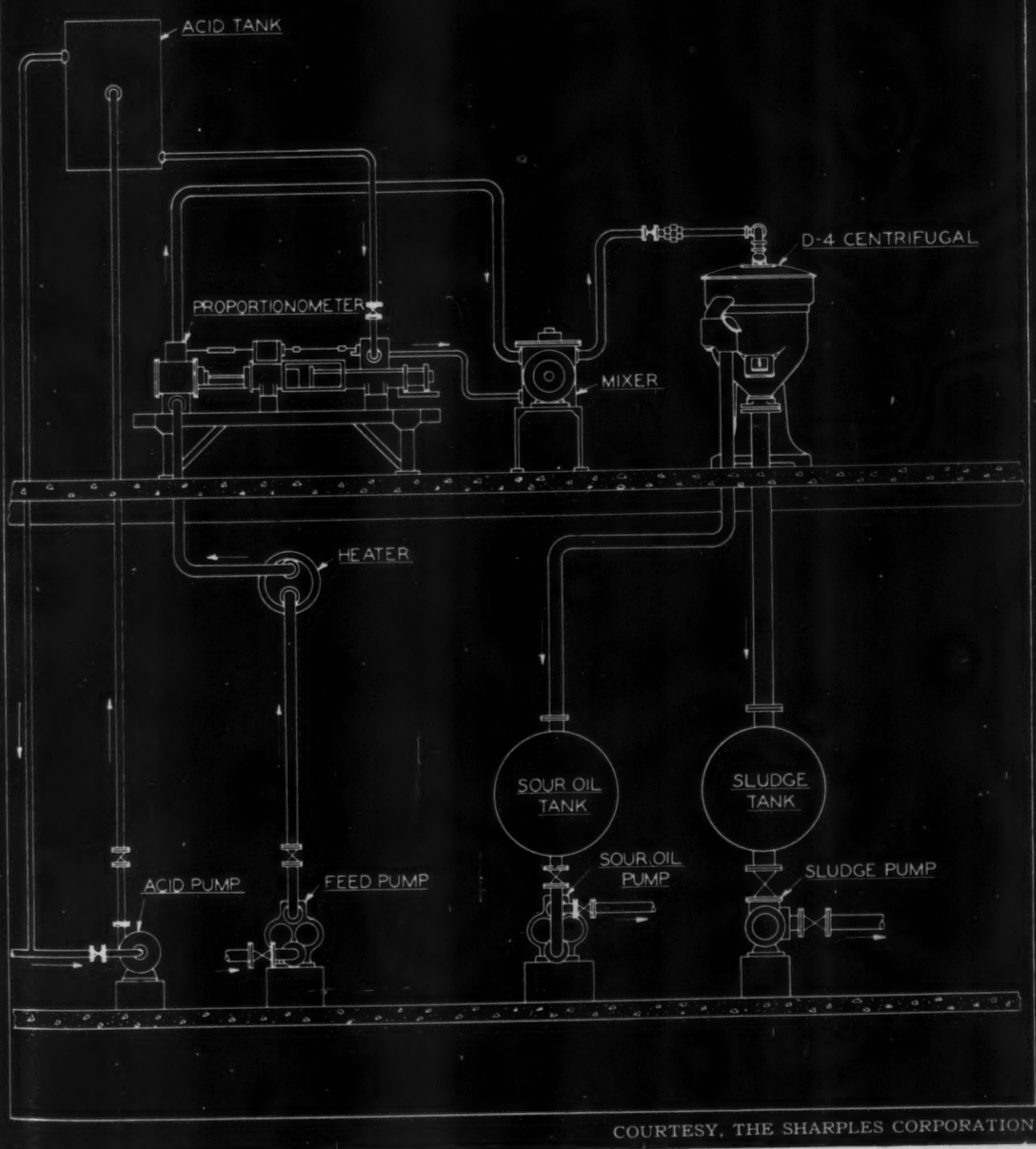
Freedom from sludge in the sour oil facilitates neutralization whether by caustic washing or contact filtration. The tendency toward formation of stable oil-caustic emulsions is reduced and contact filtering rates are materially increased.

Complete freedom from atmospheric contamination by sulfur dioxide, acid fog and hydrocarbon vapors in cracked distillate treating, is assured.

In the treatment of non-viscous oils, the low acidity of centrifugally separated oils is responsible for a reduction in neutralization reagent cost of 40-60 percent.

Oils which have been solvent extracted for V.I. improvement may be acid treated for asphalt removal and clay saving "in viscosity" when centrifugal separation of sludge is used.

SHARPLES ACID TREATING PROCESS



COURTESY, THE SHARPLES CORPORATION

Sharples Bari-Sol Dewaxing Process

THE SHARPLES CORPORATION

THE Sharples Bari-Sol Process of centrifugal dewaxing has been so named for the reason that it makes use of solvents which are heavier than either the oil to be treated or the wax to be separated therefrom.

A solvent which is sufficiently heavy to cause the wax to be lighter than the oil solution allows the wax to be withdrawn from the center of the centrifugal rotor by mechanical means as an alternative to the use of naphtha as a solvent which throws the wax to the outer surface of the rotor where a carrier liquid is used to control the continuous discharge of the wax. In consequence, a centrifugal operation employing a heavy solvent permits the centrifugal dewaxing of lubricating oil stocks of any character irrespective of the crystallinity of the wax contained in the oil.

The Bari-Sol process of centrifugal dewaxing has several advantages. Any lubricating stock may be successfully dewaxed. The stock may be dewaxed either in its raw residual state or after distillation, hydrogenation, or solvent extraction. It will handle paraffine distillates and not only produce therefrom an oil of satisfactory cold test but a wax which will readily sweat. The pour test of the finished oil closely approaches the temperature at which the oil is centrifuged. The dilute stock may be chilled at a rapid rate with a relatively large temperature differential between the oil and the chilling medium. The temperature rise of the wax-free oil solution through the centrifugals is low, which permits a maximum heat recovery and by which refrigeration requirements are held to a minimum. The operation is carried on at atmospheric pressure. The recovery of oil by rerunning the wax with additional solvent with the return of recovered oil solution to the primary dewaxing operation results in a high yield of oil and a wax of low oil content with a substantially lower installation and operating cost for refrigeration and solvent recovery than is the case in those processes using filters for wax separation, in which the wax washing solvent represents extra refrigeration and the recovered oil solution extra solvent recovery.

The solvent employed is a mixture of ethylene dichloride and benzol, stable and non-corrosive, and there has recently been developed for the process a solvent mixture which is non-inflammable.

The flow diagram on the opposite page shows the diluting, chilling and centrifugal separating steps of the Bari-Sol process.

A description of the manner in which the process operates follows: Stock, solvent and recovered oil dilution (from the rerun centrifugals) are blended in a homogeneous mixture in the heating and blending tank, which is provided with suitable agitator and steam coils for heating, if necessary. The blended stock and solvent dilution, in proportion of approximately three parts of solvent to one of stock, is then charged in series first through the exchanger, chilling machines, countercurrent to the cold dewaxed oil dilution, and then through the direct ammonia expansion chilling machines to the primary centrifugals.

The dilute dewaxed oil discharges from the primary centrifugals to a run tank and is then transferred to the solvent recovery unit at the plant capacity rate through the exchanger chilling machines countercurrent to the processing stock for the recovery of refrigeration.

The waxy stream discharged from the primary centrifugals flows to a run tank from which it is charged at the plant capacity rate to the rerun or secondary centrifugals. At the ends of the rundown header to the run tank cold solvent make-up is added in order to assist the flow of the waxy discharge to the run tank and to increase the solvent to waxy oil ratio. The run tank is provided with a slow-speed wide-sweep agitator to insure a uniform blend of the waxy discharges from the centrifugals and the make-up solvent.

The recovered oil solution discharge from the rerun centrifugals flows to a run tank and from here is discharged to the blending tank. On the way to the blending tank the recovered oil solution is exchanged with the solvent make-up of the rerun centrifugals through a shell and tube type exchanger for recovery of refrigeration.

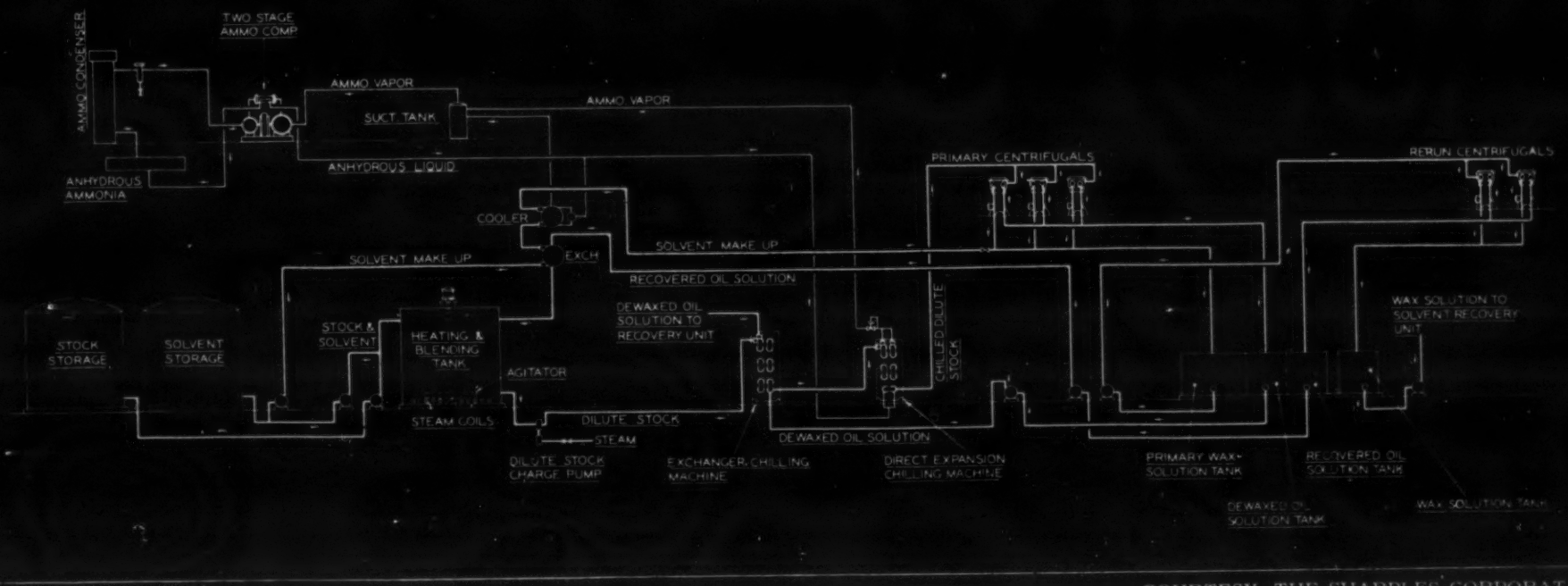
The wax solution discharged from the rerun centrifugals flows into a run tank and is then transferred to the solvent recovery unit for the continuous recovery of solvent.

Provisions are made so that dewaxed oil is furnished under pressure to the gland seal rings of the chilling machines and of pumps handling solvent or solvent dilution so that there will be no loss of solvent in leakage from this source.

The photograph insert on the opposite page shows a number of the centrifugals in the installation of the Bari-Sol process at the Ponca City, Oklahoma, refinery of the Continental Oil Company.

This description of the Sharples Bari-Sol Process and the flow chart on the opposite page are presented through the courtesy of The Sharples Corporation, Twenty-third and Westmoreland Streets, Philadelphia, Pennsylvania.

THE BARISOL PROCESS



COURTESY, THE SHARPLES CORPORATION

Sharples Naphtha Dewaxing Process

THE SHARPLES CORPORATION

THE SHARPLES PROCESS of centrifugally dewaxing lubricating oils has been in commercial operation for 26 years, during which time over 50 plants have been put into operation in this and several foreign countries.

This process is used for the separation of amorphous waxes from residual and heavy overhead stocks. It has always been considered the simplest and most economical method of dewaxing. As a result of further research on the Sharples Process a new technique has been developed which further strengthens the economic claims of this method of dewaxing.

The new development makes possible increased yields of dewaxed oil in existing plants and maximum yields of dewaxed oil in new installations. No special solvent is required other than the naphtha, present in any refinery, and normally used in the Sharples Process. The new technique also lends itself readily to the production of high melting point waxes. In all cases the addition to an existing plant can be made with little or no interruption to present operation.

The new technique involves the recentrifuging of the petrolatum from the normal dewaxing operation. It reduces the amount of naphtha used for primary separation so that the maximum requirements are two parts of naphtha to one of stock. The petrolatum produced as a result of the primary operation is then diluted with approximately half of the naphtha that is used in the primary operation, equivalent to a dilution ratio of five or six parts of naphtha to one of the primary petrolatum, and recentrifuged for oil recovery. The recovered oil solution coming from the secondary operation is then used as a dilution makeup for the primary operation. Thus the process permits the use of the minimum amount of dilution at the point where cold test and cloud are being made and the maximum amount of dilution at the point where the oil is being recovered from the petrolatum for increasing yield. Total naphtha needed for the dewaxing operation is not increased but frequently is decreased.

In an existing plant where it is desired to add the secondary operation the present plant need not be disturbed except for the final "tie-in" of the secondary system unless it would be profitable at the same time to change the chilling system so that a maximum of refrigeration could be recovered by exchange. The incorporation of such savings in refrigeration minimizes the additional refrigeration requirements for the added secondary operation and often enough saving of refrigeration can be effected to completely take care of the secondary operation.

In a new plant the primary operation would consist of a small blend tank where the stock and naphtha are blended in approximately a 1:1 dilution. This solution is pumped through a vertical exchanger chilling tower where it is chilled by dewaxed oil solution to about 85° F. At this point the recovered oil dilution is added and the chilling in full dilution is continued further by exchange and finally by direct expansion ammonia to the required centrifuging temperature. From the chillers the cold solution is fed at constant pressure to the primary centrifuges where it is separated into dewaxed oil and primary pet.

The dewaxed oil on its way to the naphtha reduction unit, is pumped continuously through the exchanger system for maximum recovery of refrigeration.

The oily pet should preferably be dehydrated by partial or complete removal of the naphtha in order to eliminate ice in the chilling system. If it is desired to separate high melting point wax this moisture removal step will be unnecessary in that the moisture will be removed in the high temperature centrifuging stage. The primary oily pet is blended with five or more volumes of naphtha, equivalent to approximately one half of the primary dilution requirements. This blend is then rapidly chilled to the same temperature under which the primary centrifuging was carried out by pumping through large diameter double pipe scraped surface exchanger chillers and similar type direct expansion chillers. The cold solution is then fed at constant pressure to the secondary centrifuges where it is separated into recovered oil and petrolatum.

The recovered oil is passed continuously through the secondary exchanger chillers enroute to enter the primary chilling system as described previously.

To add still further to the economy of the process a new Sharples D-4 centrifuge has been perfected which has four times the capacity of the centrifuge previously furnished for this service. This centrifuge can be utilized in primary or secondary service.

In installations where conditions warrant it, the secondary system may be varied to produce high melting point waxes. The melting point of the wax depends entirely upon the nature of the stock and the temperature at which they are separated. Thus to provide for this step it would only be necessary to bleed out of the secondary chilling system at any desired temperature, and centrifuge. The high melting point wax is recovered from the system and the remaining low melting point wax returned to the chilling system at the same point from which it was originally removed for further chilling to the temperature of the normal secondary operation for recovery of oil.

This description of the Sharples Process and the flow chart on the opposite page are presented through the courtesy of The Sharples Corporation, Twenty-third and Westmoreland streets, Philadelphia, Pennsylvania.

SHARPLES DEWAXING PROCESS
COURTESY, THE SHARPLES CORPORATION

Shell Butane Isomerization Process

SHELL DEVELOPMENT COMPANY

THE greatly expanded alkylation capacity, brought about over the past five years by the unprecedented wartime demand for high-octane aviation gasoline, has been largely maintained in operation to meet peacetime upgrading of the octane number of motor fuels. This postwar production of alkylate requires a high consumption of isobutane, used in its manufacture, which in many refineries is greater than the amount normally available from natural gasoline and cracking operations. The Shell Butane Isomerization Process provides means for supplying this deficiency.

Briefly, the process is one in which normal butane is converted, under the influence of a catalyst, to isobutane. In many naturally occurring mixtures of butanes, or synthetically prepared cuts of butylenes and butanes, the proportion of normal butane is greater than that of isobutane.

A typical analysis of a C_4 cut from reforming operations (i.e., the fraction containing butanes and butylenes) is as follows:

	Percent by Weight
Isobutylene	20.0
Normal butylenes	27.0
Isobutane	18.5
Normal butane	34.5
	100.0

Such a mixture is not suitable as the sole charge stock for alkylation operations as it is deficient in isobutane. However, if normal butane, which usually is discarded from the operation, is taken as charge stock for an isomerization unit, the requisite quantity of isobutane will be formed for charging to an alkylation unit. Some of the normal butane charged to the isomerization unit is unconverted and accompanies the isobutane. The unconverted normal butane is segregated in the fractionation section of the alkylation plant and is once more charged to the isomerization unit, thus the normal butane is recycled through isomerization and alkylation plants until conversion is completed, eliminating or materially reducing the necessity for obtaining natural isobutane.

The process consists of passing normal butane in vapor form, at a moderate temperature, through one or several reactors, each containing a granular bed of catalyst, a constituent of which is aluminum chloride. Dry hydrogen chloride gas is incorporated with the butane vapors to act as a catalyst promoter. The construction of the reactors may provide for a single catalyst bed, or may be of a heat-exchanger type with the catalyst placed in tubes surrounded by circulating cooling liquid as shown on the accompanying diagram.

The discharge from a single pass through the reactors flows to a receiver through a cooler and a refrigerated condenser which condenses all the butanes, the hydrogen chloride going into solution. A small amount of uncondensable gas is vented from the receiver after being scrubbed with product recycle to recover hydrogen chloride, and the condensate is charged to an HCl stripping column, which effects substantially complete removal of recycle hydrogen chloride gas.

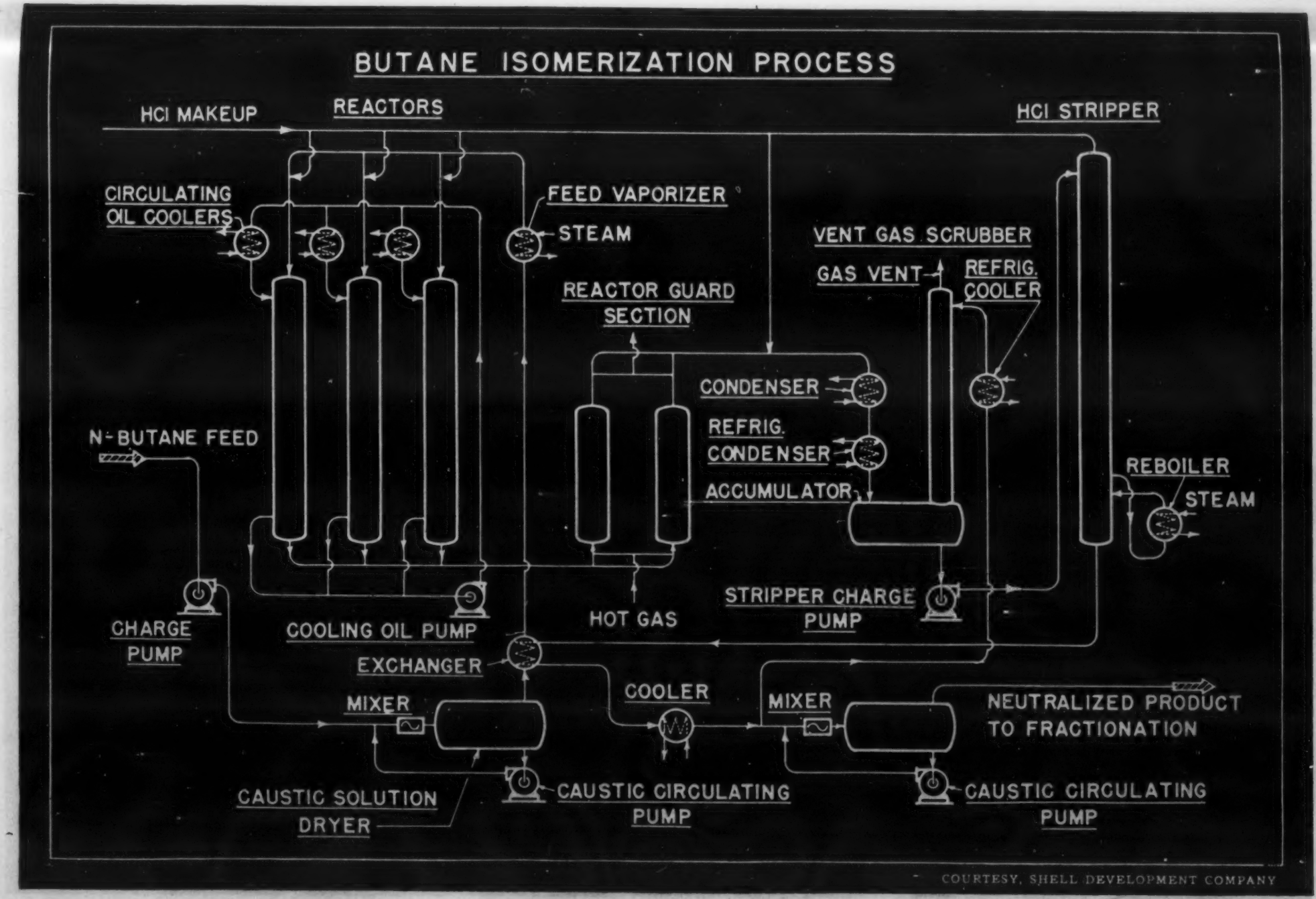
The accompanying flow sheet shows the essential features of a typical isomerization plant. The hydrocarbon charge is picked up by the charging pump and passed through a drying step which extracts the last trace of moisture. The feed is completely vaporized and the flow is divided in the desired proportions to the reactors; each individual stream receives its correct amount of hydrogen chloride recycle gas.

The vapors pass through the catalyst beds and exit streams combine before flowing through coolers and condensers. The condensate is run to a receiver from which it is pumped to the HCl stripping column. Butanes, substantially free of hydrogen chloride, flow from the base of the column, while vapors, consisting largely of hydrogen chloride, are recycled to the reactors. The last traces of hydrogen chloride in the isomerized product are removed by caustic scrubbing.

No special materials are required for the construction of the plant since dry hydrogen chloride, under the conditions prevailing in the process, is not corrosive. Temperatures and pressures under which the plant operates are moderate, consequently the required capital outlay is low.

The catalyst has been developed specially for the purpose. It is rugged, cheaply produced, and has long life; these factors combined make its regeneration unnecessary. The catalyst is resistant to poisons, but it is necessary to reduce the proportion of olefins and pentanes in the charge stock to a low figure, as both of these produce objectionable by-products and tend to shorten the life of the catalyst.

The process is licensed by Shell Development Company, New York.


BUTANE ISOMERIZATION PROCESS
HCl MAKEUP
REACTORS
HCl STRIPPER
CIRCULATING OIL COOLERS
FEED VAPORIZER
STEAM
VENT GAS SCRUBBER
GAS VENT
REFRIG. COOLER
REACTOR GUARD SECTION
CONDENSER
REFRIG. CONDENSER
ACCUMULATOR
N-BUTANE FEED
REBOILER
STEAM
CHARGE PUMP
COOLING OIL PUMP
HOT GAS
STRIPPER CHARGE PUMP
EXCHANGER
COOLER
MIXER
MIXER
NEUTRALIZED PRODUCT TO FRACTIONATION
CAUSTIC SOLUTION DRYER
CAUSTIC CIRCULATING PUMP
CAUSTIC CIRCULATING PUMP
COURTESY, SHELL DEVELOPMENT COMPANY

Shell Catalytic Hydrogenation

SHELL DEVELOPMENT COMPANY

A SIMPLE and economical catalytic hydrogenation process has been developed by the Shell Companies and used successfully for several years for hydrogenation of octenes produced by polymerization of butylenes. The process operates at very mild conditions of temperature and pressure, effecting substantially complete hydrogenation and producing fuels low in acid-heat number ideally suited for manufacture of high-octane aviation gasoline.

Commercial application to date has been limited to direct hydrogenation of an octene mixture prepared by the butylene polymerization process described in this volume under the title "Hot Acid Polymerization." The product of these two processes is a saturated octane of 98 to 100 octane number. The hydrogenation process is by no means limited to treatment of the octene mixture described here.

The mild conditions under which this process operates are made possible by the use of a nickel catalyst supported on porcelain. The unformed catalyst is a hydrated nickel nitrate impregnated in porcelain. The nitrate is decomposed to nickel oxide and then reduced to metallic nickel by a stream of hydrogen. After formation the catalyst is maintained in an atmosphere of hydrogen when not in use. Oxygen and carbon monoxide cause temporary loss of activity which can be restored by regeneration in place. Sulfur compounds cause permanent poisoning; thus, the sulfur content of the octene feed must be held to a minimum by proper treatment of the butylenes before polymerization. Spent catalyst can be used for an additional pre-treatment of the octenes to prevent premature catalyst poisoning. A catalyst life of 1000 gallons of finished iso-octane per pound of nickel is common.

Hydrogen for plant operation should be free from sulfur; treatment before use may be necessary. Convenient sources of hydrogen are the pyrolysis of natural gas and dissociation of ammonia. A cheap process for production of hydrogen, free of impurities which poison nickel catalysts, has been developed and used for some time by the Shell Chemical Company. This gas contains only small amounts of methane and nitrogen as impurities, both of which are inert and not objectionable in these concentrations.

When the activity of the catalyst decreases to a certain point, it is reactivated in place. Although initial activity is not restored, two to four reactivations lengthen catalyst life by 150 percent. Activity declines slowly after each reactivation, and, ultimately, the catalyst must be removed from the converters and regenerated.

Figures 1, 2, and 3 on the page opposite indicate the type of equipment used. To facilitate removal of the heat of reaction the catalyst is packed in vertical tubes arranged in bundles similar to tube and shell heat exchangers. A circulating fluid occupies the space around the tubes automatically maintaining the reaction temperature. For catalyst formation and reactivation, which take place at higher temperatures, a separate, hot-fluid, circulating system including a furnace is used. When several converters are in use it is possible to hydrogenate in some chambers while other catalyst beds are being used as guard chambers and still others are being formed or regenerated.

The hydrogen heater and octene vaporizer are heated by a hot fluid. Heat exchange is available even in the single stage system.

Deterioration of equipment by corrosion is negligible. Because of low temperature and pressure no special alloys or other materials are required.

Operation and control of hydrogenation plants are simple. The primary control point is the degree of unsaturation of the finished product as determined by titration with a weak brominating agent.

Use of catalyst guard tubes is advantageous for sulfur removal and catalyst conservation. Ordinarily the guard catalysts are not effective in promoting hydrogenation themselves. All the hydrogen feed may be fed into the octene vapors prior to passing through the guard tubes, but this large amount of hydrogen is not necessary for effective sulfur removal. Therefore, the major portion of the hydrogen feed may be introduced following the guard tube, thereby increasing the contact time of the octenes in the guard tube chamber. Roughly one volume of guard catalyst is sufficient for two volumes of active catalyst. Some plants contain a separate catalyst tube for the hydrogen feed to convert traces of carbon monoxide to methane and to preclude formation of nickel carbonyl on cold spots in the catalyst bed. This is seldom necessary.

The presence of water is not injurious to the catalyst; steam is usually used to purge the system of either air or hydrogen.

This process has been developed by Shell Development Company, New York.

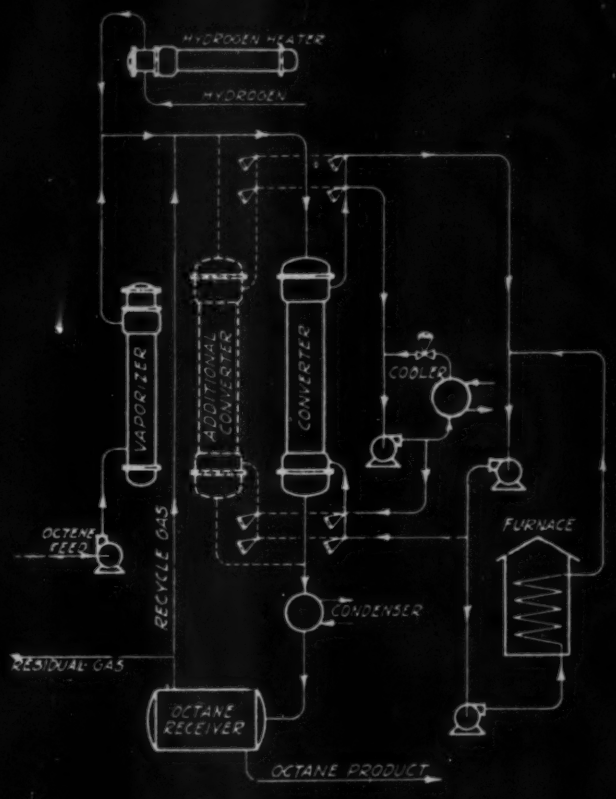


Figure 1—Single-Stage System

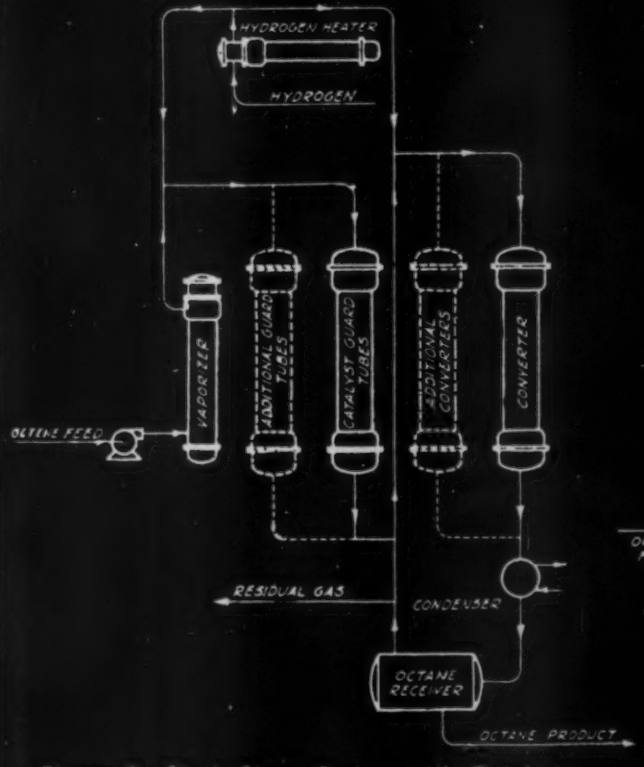


Figure 2—Single-Stage System with Catalyst Guard Tubes

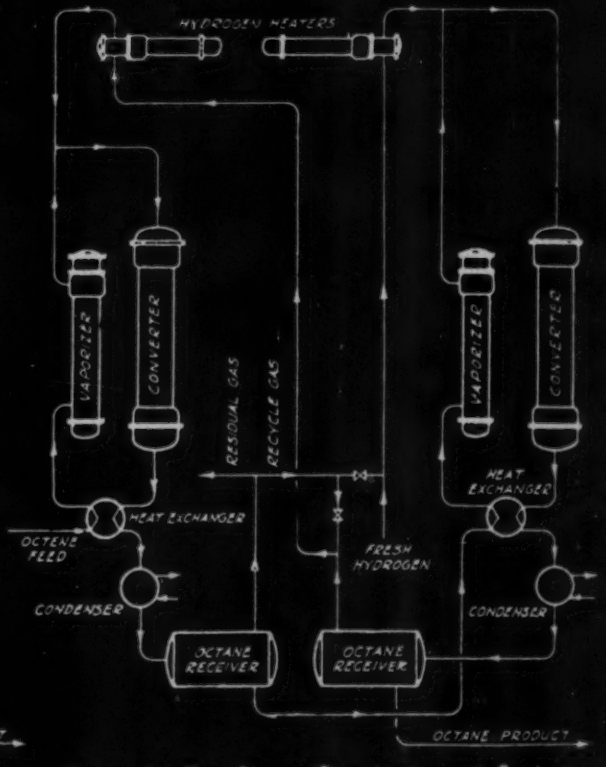


Figure 3—Two-Stage Countercurrent System

SHELL CATALYTIC HYDROGENATION PROCESS

COURTESY, SHELL DEVELOPMENT COMPANY

Shell Liquid-Phase Isomerization Process

SHELL DEVELOPMENT COMPANY

THE Shell Liquid-Phase Isomerization Process is an entirely continuous regenerative process adaptable to the isomerization of either butane or pentane and commercial plants have proven completely satisfactory with both feeds. Some special merits of the process are: a constant high conversion for either hydrocarbon per pass coupled with a very low catalyst consumption, high ultimate yields of the desired isoparaffins, constant operating conditions, continuous catalyst make-up with continuous spent catalyst rejection, a high tolerance for feed impurities, and employment of standard fabricating methods for plant construction.

The process, in brief, consists of contacting n-butane or n-pentane in the liquid state with a catalyst comprising a solution of aluminum chloride in molten antimony trichloride. Dry hydrogen chloride gas is introduced into the feed hydrocarbon as catalyst promoter. In addition, when the process is utilized for pentane isomerization, hydrogen is introduced along with the hydrogen chloride to suppress disproportionation reactions which, if not controlled, would result in a low ultimate yield of isopentane and high catalyst consumption. The same plant can be employed for processing either of the aforementioned hydrocarbons by including a compressor to recycle the hydrogen required in the case of pentane isomerization. However, if a source of hydrogen at the desired pressure is available, such that it can be used on a once-through basis, even the compressor can be eliminated.

The simplified diagram on the opposite page shows the essential pieces of equipment and indicates the flow of the catalyst, hydrocarbon, activator and disproportionation inhibitor streams.

The normal liquid paraffin (butane or pentane) is charged continuously at essentially atmospheric temperature through one of two feed dryers, which are used alternately and renenerated periodically, through a heater where the temperature is raised to about 180° F., thence through a packed catalyst scrubber column and into the contactor. A liquid-liquid extraction of a small side-stream of the contactor catalyst with the feed takes place in the scrubber. The valuable active catalyst components pass back to the contactor in solution while the fluid inactive $AlCl_3$ hydrocarbon complex, which forms as a consequence of the small amount of side reactions, runs to the bottom of the column and is periodically drained off.

The contactor is a vertical autoclave equipped with a mixing device to provide intimate contacting of catalyst and hydrocarbon. A relatively short residence time, of the order of 10-15 minutes, in the contactor is sufficient to achieve a high conversion; 50 percent or better for butane and 55-60 percent for pentane. After phase separation of isomerizate from catalyst in the baffled upper settling section of the contactor, the isomerizate flows to the catalyst-removal column for separation of the dissolved catalyst by a simple fractionation. This recovered catalyst is pumped back to the contactor while the isomerizate is taken overhead, cooled, and passed to the accumulator from which it is pumped to the HCl stripper where the acid gas is removed and recycled to the contactor. The stripper bottoms are essentially free of HCl and after a caustic wash pass to subsequent operations.

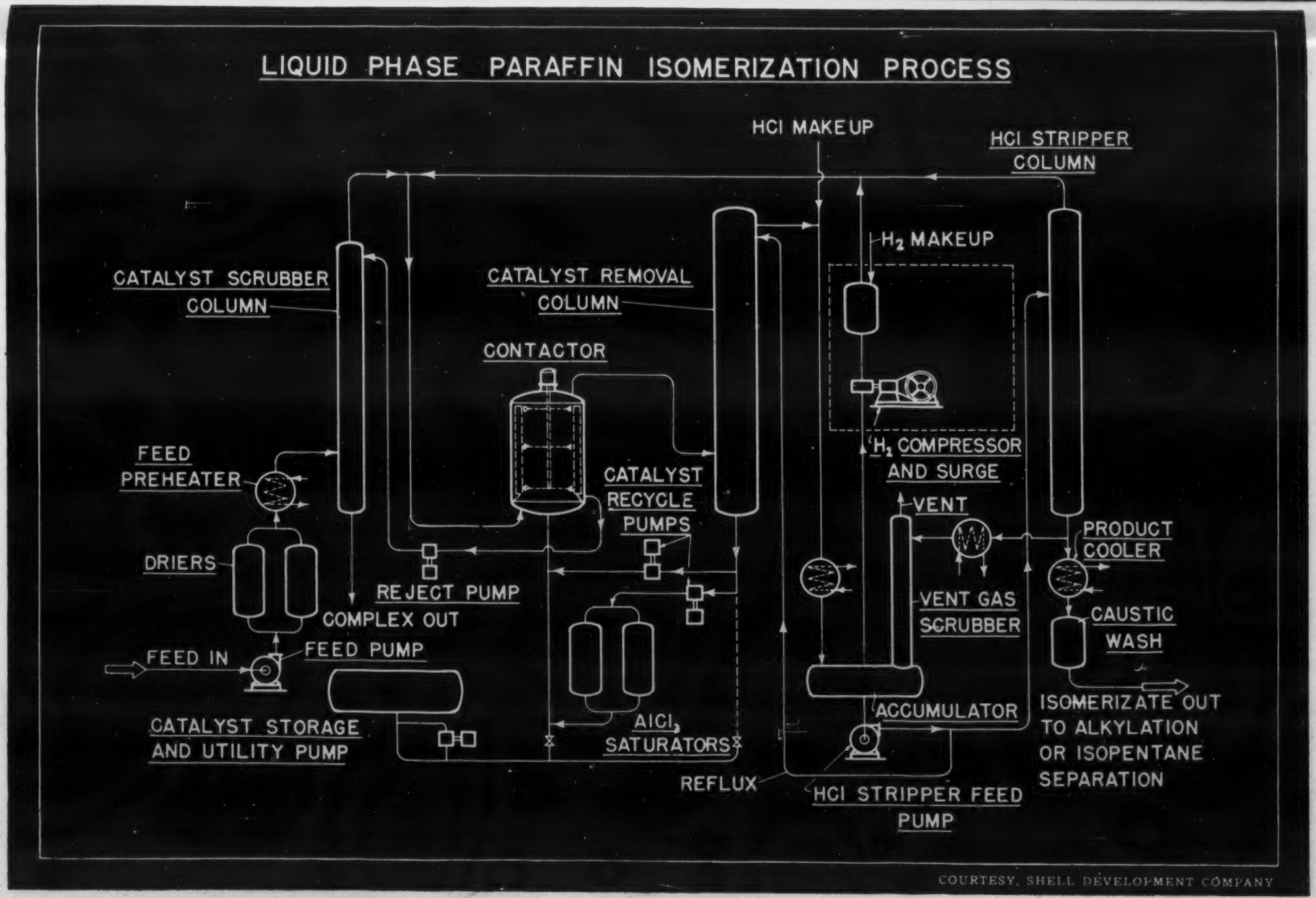
In pentane isomerization the bulk of the hydrogen is recycled from the vapor phase of the catalyst column accumulator, however, some passes in solution to the HCl stripper and is recycled with the HCl.

The small amount of uncondensable gas that is formed is vented from the vapor phase of the catalyst column accumulator through a scrubber, countercurrent to a stream of HCl stripper bottoms which reabsorb HCl and reduce its loss to a minimum.

The constant high conversion, inherent to the process, is achieved by the continuous removal of spent $AlCl_3$ complex and regeneration of the contactor catalyst with fresh aluminum chloride. To accomplish the $AlCl_3$ addition a small portion of the bottoms from the catalyst removal column, which contain only a small amount of $AlCl_3$, is pumped through one of two $AlCl_3$ saturator vessels, filled with lump $AlCl_3$, before it is returned to the contactor. Since the $AlCl_3$ consumption in the process is quite low, this procedure serves to maintain the required concentration.

Since the catalyst is solid at atmospheric temperatures, lines carrying it are heat traced. Above its melting point, 165° F., the catalyst is quite fluid and easily transferred by pumping. All major pieces of equipment are of steel except those handling the catalyst which are nickel-clad. The temperatures of operation in the various units are moderate while the highest pressure necessary (HCl stripper) is around 325 psig.

This process is licensed by Shell Development Company, New York.



COURTESY, SHELL DEVELOPMENT COMPANY

Shell Phosphate Process

SHELL DEVELOPMENT COMPANY

THE Shell Phosphate Process is a regenerative liquid purification system for the removal and recovery of hydrogen sulfide from gaseous and liquid hydrocarbons. The process consists essentially of two steps: absorption or scrubbing with a solution of tripotassium phosphate and regeneration of the spent solution. The principal absorption reaction is expressed

$$H_2S + K_3PO_4 \rightarrow KHS + K_2HPO_4$$

Regeneration reverses this reaction.

The hydrogen sulfide recovered is essentially free of hydrocarbons and is an ideal raw material for the manufacture of sulphuric acid or elemental sulphur.

The inexpensive solution used ordinarily is prepared at the plant from commercial grades of potassium hydroxide and phosphoric acid in such proportion that the molal ratio of KOH to H_3PO_4 is between 2.95 and 3.00 to 1. The optimum concentration of K_3PO_4 depends upon the amount and nature of impurities in the hydrocarbon processed. Since K_3PO_4 is a stable inorganic salt, which does not decompose nor vaporize under treating conditions, solution loss is negligible, being confined to leakage at packing glands of pumps, etc. Such leakage can be collected and returned to the system. K_3PO_4 does not contaminate nor polymerize the hydrocarbon.

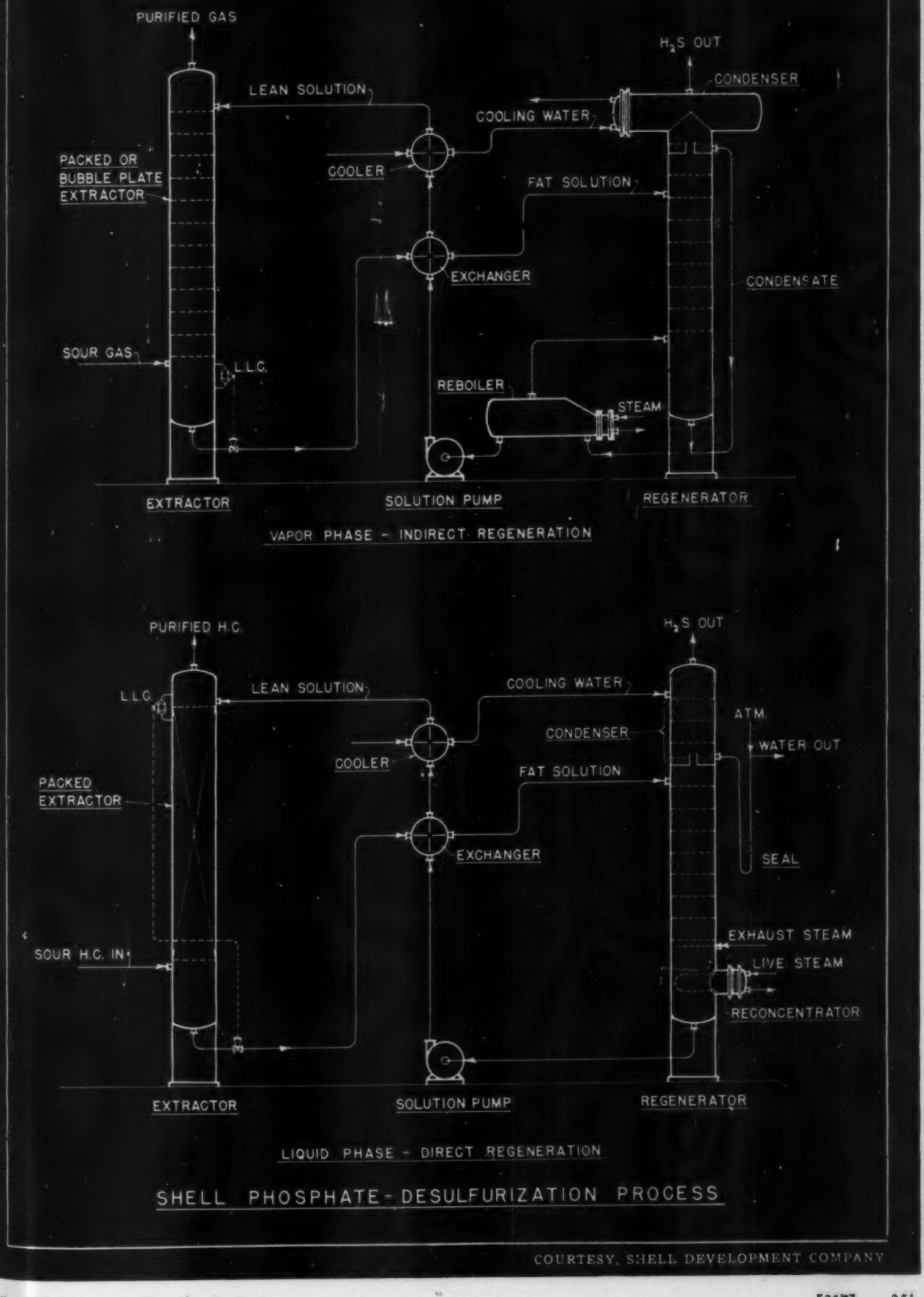
Iron and steel are satisfactory for all equipment. In case of corrosive cooling-water, Admiralty-metal tubes and Muntz-metal sheets and baffles may be used for condenser and solution cooler. Gauges, instruments, valves and pumps should be all iron and steel. Monel or stainless-steel trim can be used. Observance of these general specifications will insure freedom from corrosion on the process side.

The accompanying flow diagrams indicate the necessary equipment. Gases are scrubbed in a countercurrent absorber of bubble-plate or packed-tower construction. The hydrogen-sulfide content of gases can be reduced low enough to satisfy any reasonable refinery requirement. Treatment at temperatures as high as 200° F. is possible with the Prosphate Process.

Equilibrium of tripotassium phosphate with liquid hydrocarbons is even more favorable than with gases. A hydrogen sulfide content of treated propane-butane mixtures as low as 0.02 mol-percent is easily attained.

Regeneration of the spent solution is accomplished by heating the solution to its boiling point and stripping with steam in a bubble-plate column. Steam for stripping may be provided from the solution by heating the solution indirectly through a tube bundle, the heat being supplied by hot oil or by steam at a moderate pressure. Altenately, exhaust steam at low pres-

This process is licensed by Shell Development Company, New York.



SHELL PHOSPHATE-DESULFURIZATION PROCESS

COURTESY, SHELL DEVELOPMENT COMPANY

Shell Solutizer Process

SHELL DEVELOPMENT COMPANY

THE Shell Solutizer Process for sweetening gasolines and naphthas is unique in that a doctor-sweet product is made by substantially complete physical extraction, instead of chemical conversion, of mercaptans. For the extraction, caustic solution containing an organic substance (such as potassium isobutyrate), called for brevity Solutizer, is used. The removal of mercaptans gives products characterized without exception by an improvement in their TEL susceptibility and, very often, by an improved initial octane rating, compared to those sweetened by conventional doctor or other treatments based on the oxidation of mercaptans to disulfides. In addition, there is a reduction in total sulfur brought about by the Shell Solutizer sweetening which in some cases is sufficient to eliminate sulfuric acid treatment normally required to produce fractions conforming to low-sulfur specifications. In other cases, the Shell Solutizer sweetening applied prior to acid treating results in the reduction of acid requirements. However, even when the Shell Solutizer Process replaces doctor or other sweetening treatment of acid-treated stocks, substantial economies and improvements in product qualities are effected.

The process is applicable for sweetening either straight-run cracked, reformed, or synthetic fractions, and its effectiveness even extends to the products boiling outside the normal gasoline range.

The Shell Solutizer Process is continuous and fully regenerative. Essentially, it consists of mercaptan extraction with a lean solutizer-solution and the regeneration of the fat solutizer-solution by boiling and stripping with steam. As indicated on the accompanying flow diagram, a sour gasoline or other hydrocarbon fraction is first pre-treated with a caustic solution of suitable concentration to remove such impurities as carbonoxylic and other relatively strong acids or to adjust the concentration of some acidic components, such as alkyl phenols, to permit efficient solutizer treatment. Usually a batch of caustic solution is used for pre-treating until practically exhausted. The pretreated gasoline is passed on to a countercurrent extraction unit in which it is brought into contact with a lean solutizer-solution. Extraction columns packed with carbon Raschig rings are effective and proven for this purpose. The sour hydrocarbon fraction is introduced near the bottom of the column and the lean solutizer-solution enters near the top of the column. Intimate countercurrent contact of the two streams results, and a doctor-sweet fraction and fat solutizer-solution are withdrawn from the opposite ends of the column.

The fat solutizer-solution then is diluted with a condensate produced in the regenerating step. The diluted solution is allowed to settle in a separator, where a small quantity of sour gasoline, initially partly entrained and partly dissolved in the fat solution, separates out as the result of the dilution. This sour gasoline is skimmed and returned to the extraction column. The separated fat solutizer-solution is preheated and fed to the top plate of a bubble-plate solutizer-solution regenerator. The fat solution is regenerated by being stripped of mercaptans by steam either injected from an outside source or generated in a reboiler at the bottom of the regenerator. A temperature in the neighborhood of 270° F. is maintained at the bottom of the column; it varies somewhat with the composition of the solutizer solution and the pressure. The regenerated or lean solution is pumped through heat exchangers and coolers to the top of the extraction column.

The steam and mercaptans pass from the regenerator to the condensate stripper and on to, first, the condenser and, then, to a separator. The condensate is pumped from the latter to the top of the condensate stripper, from which a mercaptan-free condensate is withdrawn to dilute the fat solutizer-solution just prior to its entering the gasoline separator. The mercaptans separated from the condensate are available for chemical syntheses or can be disposed of as fuel.

The construction of the Shell Solutizer Process plant presents no unusual engineering or corrosion problems. With the exception of a copper-nickel-alloy tube-bundle in the reboiler, the equipment can be made of low-carbon steel.

One of the developments of the basic process permits, under certain circumstances, a treatment of several gasolines to produce their doctor-sweet blend at practically no additional cost over that of the solutizer sweetening of one of the component gasolines.

This process is licensed by Shell Development Company, New York.

SHELL SOLUTIZER PROCESS
COURTESY SHELL DEVELOPMENT COMPANY
CONDENSER
Mercaptans
Reflux
SOLUTIZER REGENERATOR
CONDENSATE STRIPPER
Fat Solution
HEAT EXCHANGER
GASOLINE SEPARATOR
Condensate
Lean Solutizer Solution
Fat Solution
SOLUTIZER EXTRACTOR
PACKED COLUMN
Doctor Sweet Gasoline
CAUSTIC PRETREATER
5% NaOH
Sour Gasoline

Solvent Dewaxing Process

TEXACO DEVELOPMENT CORPORATION

THE Solvent Dewaxing Process generally employs a mixture of dimethyl ketone (acetone) or methyl-ethyl-ketone and benzol as the solvent to obtain a dewaxed-oil pour point equal to or within a few degrees of the dawaxing temperature. The dewaxed oils are free from wax cloud and there is no rise in pour point or "pour reversion" on aging. Either distillate or residual stocks in the raw state or refined, of any viscosity, can be dewaxed. Texaco Development Corporation, 135 East 42nd Street, New York, is licensor of the process.

The process is widely used in both the United States and Europe. Thirty-two units have been installed, with a total daily charging capacity of over 52,000 barrels, processing a wide variety of lubricating-oil stocks of high and low wax content. Contracts have been awarded for 14 additional units having a total charging capacity of over 48,000 barrels per day.

Simplicity of operation, flexibility, low construction and operation costs, the absence of special equipment, special alloys, high pressure, excessively low operating temperatures and the ability to obtain high dewaxed-oil yields and very low oil-content waxes are outstanding characteristics of the process.

The wax contained in lubricating oil stocks when properly precipitated from a chilled solution in the solvent mix is particularly adaptable to removal by filtration. Practically complete separation of oil from wax is obtained by use of a cold displacement wash. The use of a closed flue-gas system minimizes fire hazards and solvent losses. Dilution solvent dosages vary from a solvent-to-oil ratio of 1.0:1 to as much as 4:1, depending upon the nature and viscosity of the charge stock.

Recently improved design of exchange and chilling equipment has resulted in reduced refrigeration requirement. Proper conditioning of the charge to the filters by controlled application of solvent has increased dewaxed-oil yield, reduced oil content of the wax to an extremely low figure, and reduced the amount of solvent circulated per barrel of oil charged. An additional development has been the production of wax, without addition agents, concurrently with the production of low-pour-point oil, but in auxiliary equipment operated as a separate process. White crystalline waxes and higher-melting-point microcrystalline waxes are produced from distillate and residual stocks, respectively, to meet specific requirements.

The accompanying flow diagram, upon which it is impractical to reflect all of the recent improvements, shows the flow through the principal chilling, filtering and solvent-recovery equipment. Dilution solvent is introduced into the charge-oil stream in such amounts, at selected points in the chilling cycle, as to insure a wax crystal structure and liquid viscosity most suitable for filtration. The chilled charge mixture flows from the double-pipe chillers through a filter-feed tank to drum-type vacuum filters in which compartmentized, cloth-covered drums rotate half submerged in enclosed filter cases. A wax-free filtrate solution is drawn through the filter cloth to filtrate tanks in which the vacuum which induces filtration is maintained. The wax cake deposited upon the drum during filtration is washed with cold solvent as it emerges from the liquid level in the filter; much of the wash solvent being drawn into the filtrate system toward the end of the filtration cycle. Just before each of the filter-drum compartments reaches the wax deflector, flue gas at low pressure is admitted through a blow-back connection to the underneath side of the filter cloth. The bond between wax and cloth is broken and the washed wax falls into the wax-discharge compartment. Controllers in the flue gas piping maintain atmospheric pressure in the filter hood.

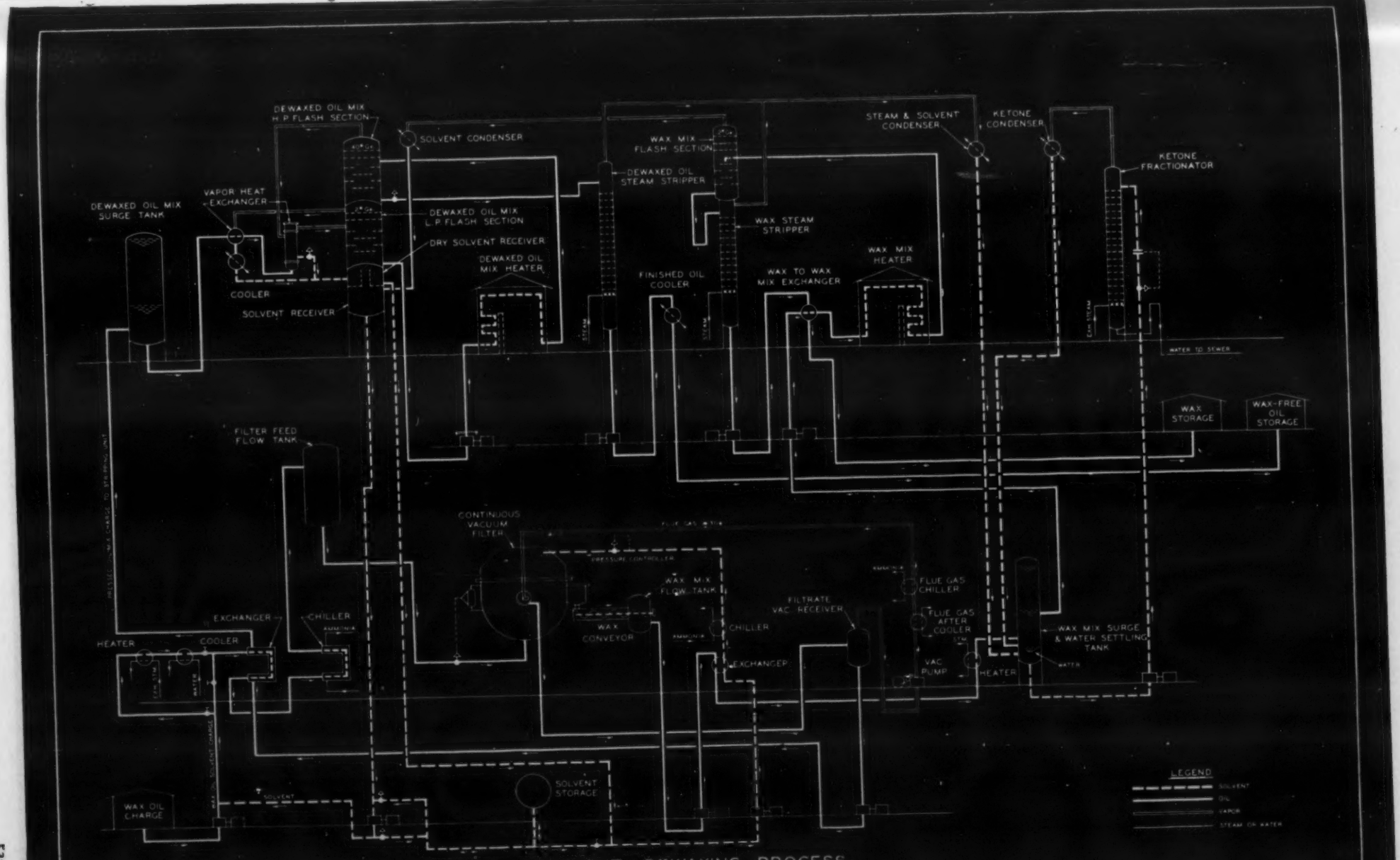
The filtrate valve on the drum trunnion is so constructed that oil-rich filtrate collected during the early portion of the filter cycle is directed to the main filtrate tank, whereas the oil-lean filtrate collected late in the cycle is directed to the wash filtrate tank. The oil-lean wash filrate then may be returned to the charge-oil stream as a substitute for a portion of the dilution solvent. Occasional warm washing of the filter cloth is a simple operation requiring 15 to 20 minutes and is conducted, without interruption to uniform flow through the plant. The warm washings, consisting primarily of solvent, generally are returned to the charge mixture, this further reducing the requirements for new solvent.

The filtrate from the main filtrate receiver is pumped through the double-pipe exchangers to evaporators for the recovery of the solvent. Heat for evaporating the solvent from the dewaxed-oil solution and wax mix may be supplied by either steam or fired heaters.

Substantially all of the water entering the system is crystallized and removed with the wax to the wax-mix surge and settling tank. This vessel is maintained at about 130° F. under which condition melted ice settles to the bottom of the tank. To this water is added condensates from steam strippers and the water-bearing material is pumped to a ketone fractionator for recovery of solvent and removal of water from the system.

The process produces high yields of low-pour-test oils free from wax cloud. Due to low solubility of wax in the solvent, low-pour tests can be obtained at relatively high separation temperatures. Extremely low pour points can be obtained, due to the narrow difference between the separation temperature and the resultant pour point.

Consistent uniformity in operating conditions and product quality is an inherent characteristic of the process. There are no corrosion or erosion problems and low-pressure equipment is used throughout.



SOLVENT DEWAXING PROCESS

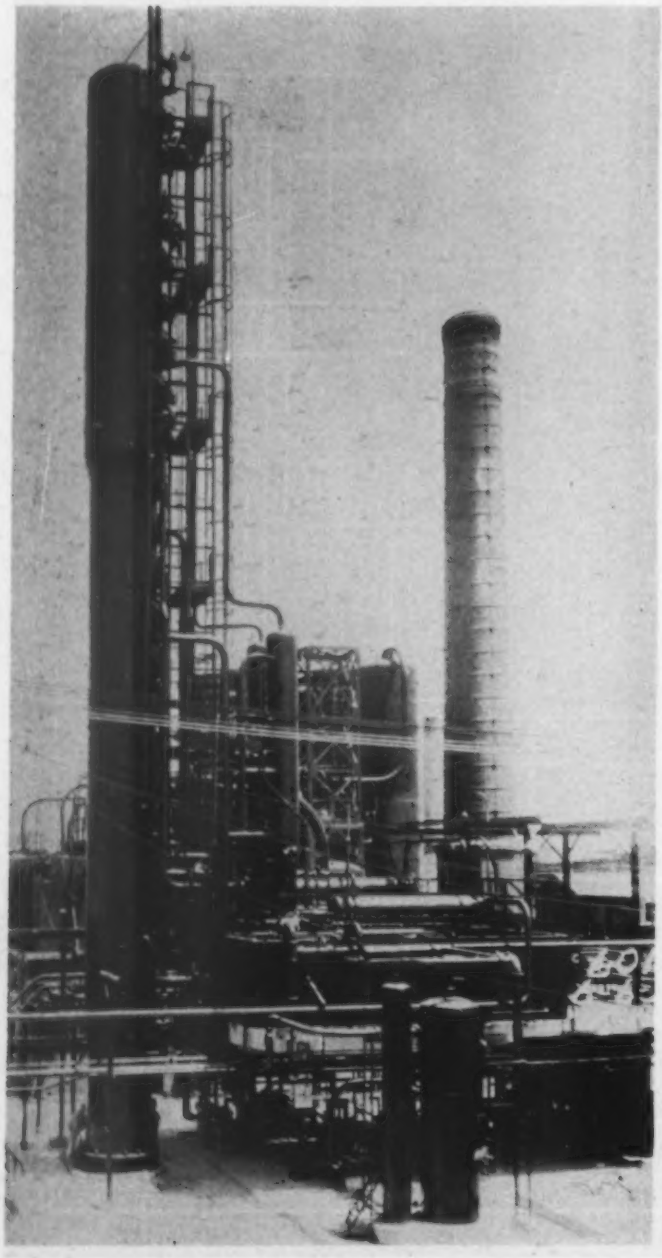
COURTESY, TEXACO DEVELOPMENT CORPORATION

Special Naphtha-Rerun Process

THE LUMMUS COMPANY

THE utility of a Lummus special naphtha-rerun unit built for a large refiner was so clearly demonstrated that the same refiner placed an order for an exact duplicate to meet his additional requirements for this service.

The engineering problem presented involved consideration of a number of factors. The chief characteristic desired was flexibility, since the unit was required to operate on a variety of naphthas and somewhat heavier distillates for production of specialty products of precise specifications. The second consideration calling for special design was introduced by the fact that material being charged to the unit had been treated and the fractions produced were to be finished products ready for marketing. For this reason it was necessary to insure that no overheating of the charge material would take place and result in off-test products. To meet these requirements a design was worked out in accordance with the flow diagram shown on the page opposite. The fractionating tower was set up for the production of an overhead stream, four side streams and a residue. The overhead product was rubber solvent or low-endpoint gasoline, depending on the characteristics of the charging stock. The four side streams were naphthas of various characteristics, most of them of narrow boiling range and well fractionated. The original intention was to produce as finished products the first and third side-stream fractions only, since the charging stock indicated that simultaneous production of more than two side streams would not enable specifications to be met. However, the performance of the unit was so much better than expectations that it was possible to produce three simultaneous side streams to specifications instead of two.



Avoidance of overheating was obtained by careful attention to the oil-heating unit. The Lummus oil heater lends itself to satisfactory solution of this problem because of its symmetry and because of the distribution of heat absorption. In view of the fact that complete vaporization of most of the various charging stocks takes place at a temperature below 450° F., it was logical to reverse normal flow of material through the heater by passing it first through the high-heat input section of radiant tubes, then through the semi-radiant roof tubes and finally through the convection bank where vaporization was allowed to take place. By the use of this flow the temperature distribution was ideal and it was possible to effect complete vaporization of the charging material without reaching a peak temperature which would be injurious to the material being handled. Further, the maximum temperature required for complete vaporization is so low that there is no sacrifice of furnace efficiency when using the reversed flow described.

Another feature of this unit which may be mentioned involves the grouping of control and metering instruments so that operation of the unit can be carried out with very little supervision and effort. To accomplish this, controls were conveniently grouped in the control room. Provision for visual inspection and flow control of all streams with remote control valves on stripping steam to the side stream strippers was provided. This arrangement was made so compact that essentially "arm-chair operation" is possible. In view of the many different services which this unit is called on to perform, the importance of close control is evident, and its value has been clearly demonstrated by the successful operation of the unit.

LUMMUS SPECIAL NAPHTHA RERUN UNIT
COURTESY, THE LUMMUS COMPANY.
VENT
TREATED NAPHTHA CHARGE
AVIATION GASOLINE
STEAM
STEAM
SPECIAL NAPHTHAS
STEAM
STEAM
SPECIAL NAPHTHAS
STEAM
BUBBLE TOWER
BOTTOMS

Superfractionation Process

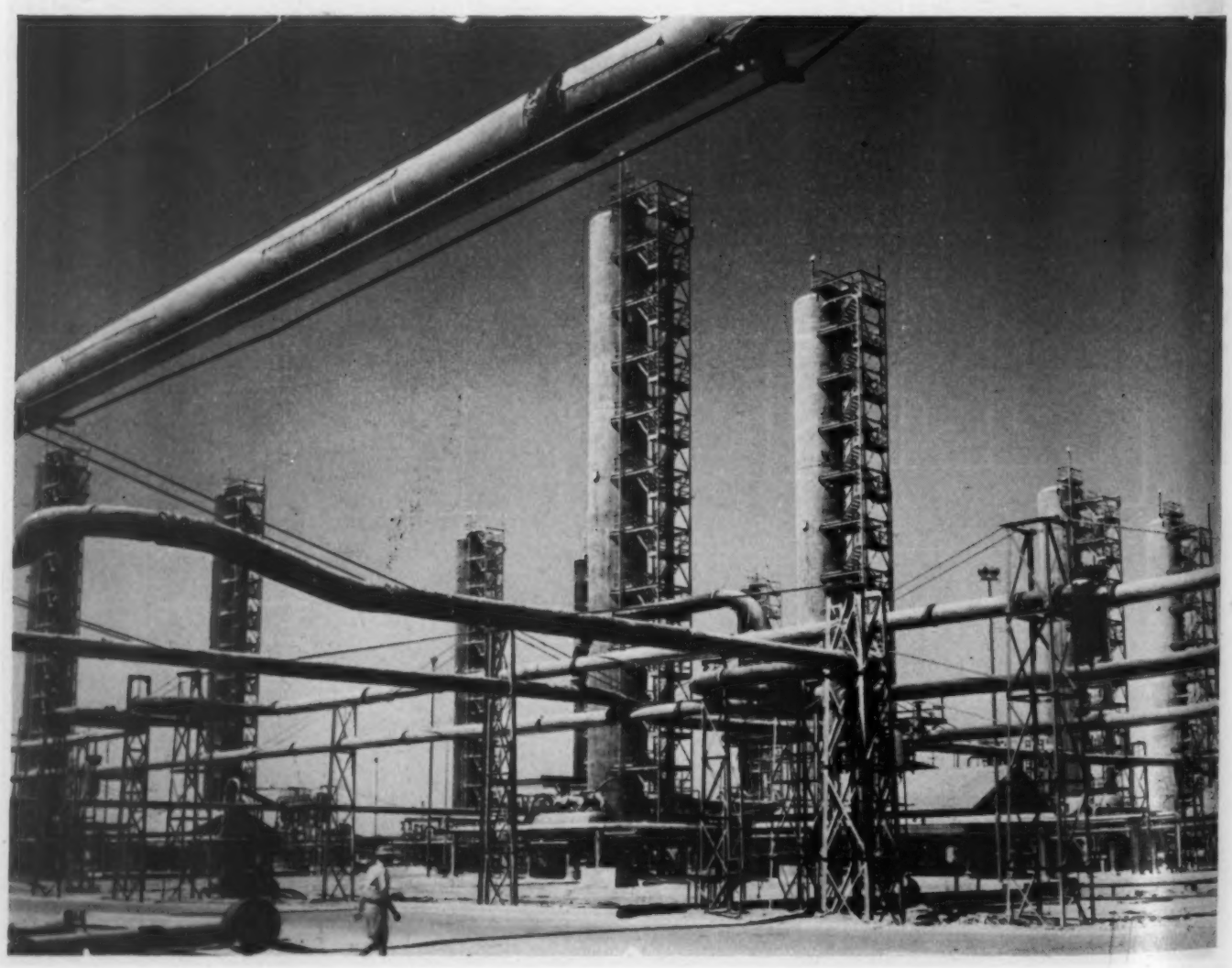
E. B. BADGER & SONS COMPANY

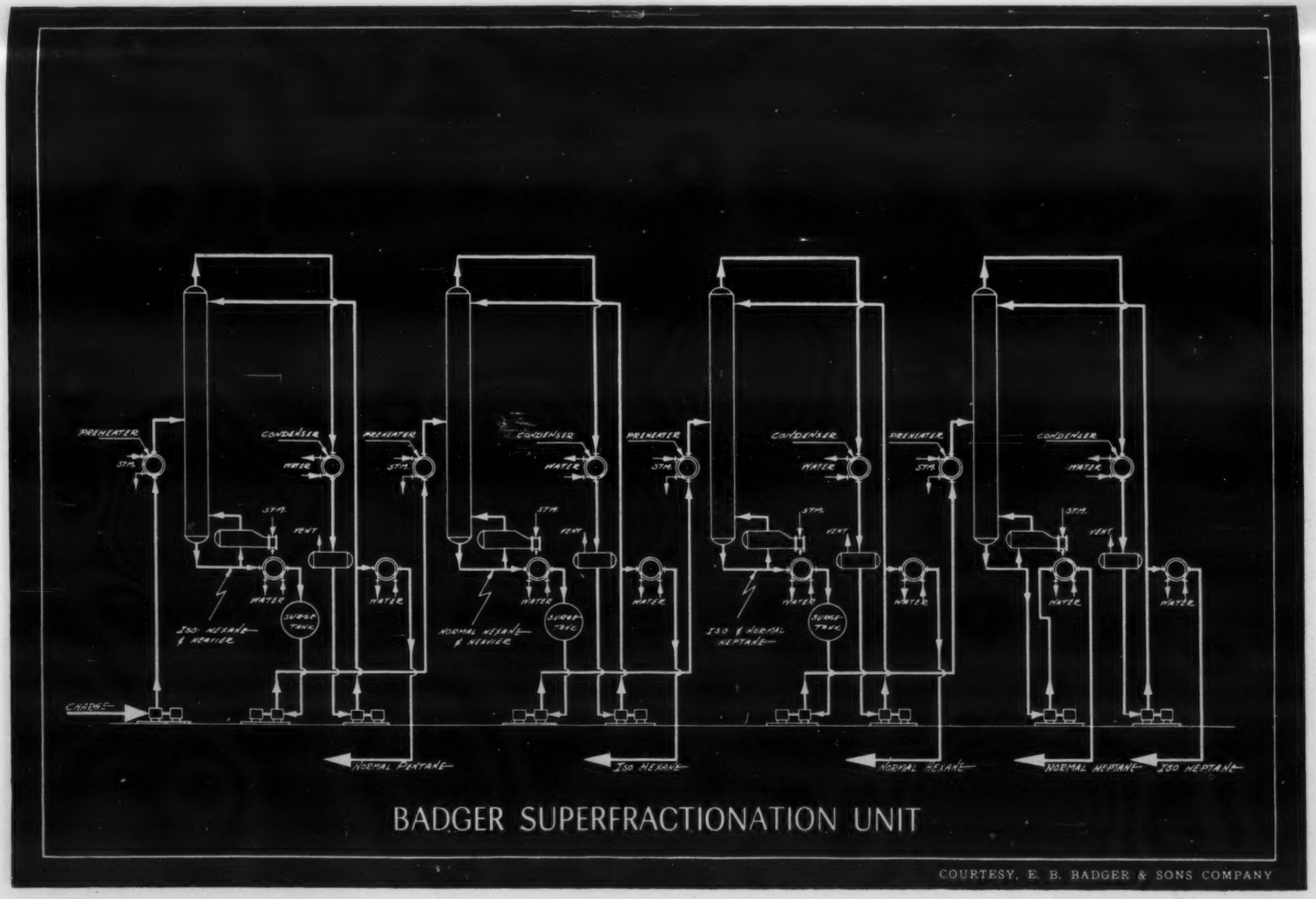
SUPERFRACTIONATION of naphtha fractions to separate the high-octane branched-chain components from the low-octane components was one method developed during the war to produce aviation blending stocks. In many instances the investment in money and steel required for aviation production by this method was but a small fraction of that required for the manufacture of high-octane materials by other means. This manufacturing procedure also may be applied to the peacetime production of high-octane motor gasolines.

In one installation by E. B. Badger & Sons Company, as illustrated by the accompanying flow diagram, a naphtha cut containing compounds ranging from normal pentane through normal heptane was charged to a series of superfractionators. Normal pentane was taken overhead in the first column and the bottoms containing isohexane through normal heptane was charged to the second superfractionator. Here isohexane was obtained as an overhead product, and the bottoms, charged to a third column, were split into normal hexane overhead, and isoheptane and heavier as bottoms. The isoheptane was taken as the overhead in the last column.

In some cases, due to the large number of plates and the resultant height, a fractionating column was split into two sections, with the vapor from the first section passing into the base of the second section, and with reflux pumped from the base of the second section into the top of the first section. In the flow sheet, for simplicity, the columns are shown as single units.

Accurate control of the distillation conditions is essential to the success of fractionation operations of this type, and Badger engineers developed the special instrumentation required. The use of surge tanks of ample capacity between the different columns contributes to steady flow and consistent operation when the composition of the feed varies. Due to the close boiling points of the fractions to be separated, product drawoff quantities generally are regulated by volume control, based on laboratory analyses of the feed stocks.





BADGER SUPERFRACTIONATION UNIT

COURTESY, E. B. BADGER & SONS COMPANY

Tannin-Solutizer Process

SHELL DEVELOPMENT COMPANY

MERCAPTAN removal processes in general may be classified into two types: those that effect sweetening by conversion of the mercaptans to other sulfur compounds, usually disulfides, and thereby result in little, if any, reduction in sulfur content; and those that achieve a physical or chemical removal of the mercaptans and substantially an equivalent reduction in the total sulfur content of the hydrocarbon treated. The Tannin-Solutizer Process is a member of this latter group and thus obtains the product improvement and increase in octane number and lead susceptibility to be derived from sulfur reduction. The process attains this objective by addition of a simple and efficient tannin-catalyzed oxidation regeneration step to a mercaptan extraction operation employing strong caustic solutions containing mercaptan solubility promoters, termed "solutizers," such as isobutyric acid and/or alkyl phenols.

The process, as illustrated in the flow diagram, consists essentially of three sections: the caustic pretreat, solutizer treatment, and regeneration system. Caustic pretreatment usually is required for removal of strongly acidic components, such as hydrogen sulfide, and in some cases for control of alkyl phenol concentration. A single conventional mixer-settler contact stage is normally satisfactory for this purpose, and the caustic solution may be used until essentially saturated.

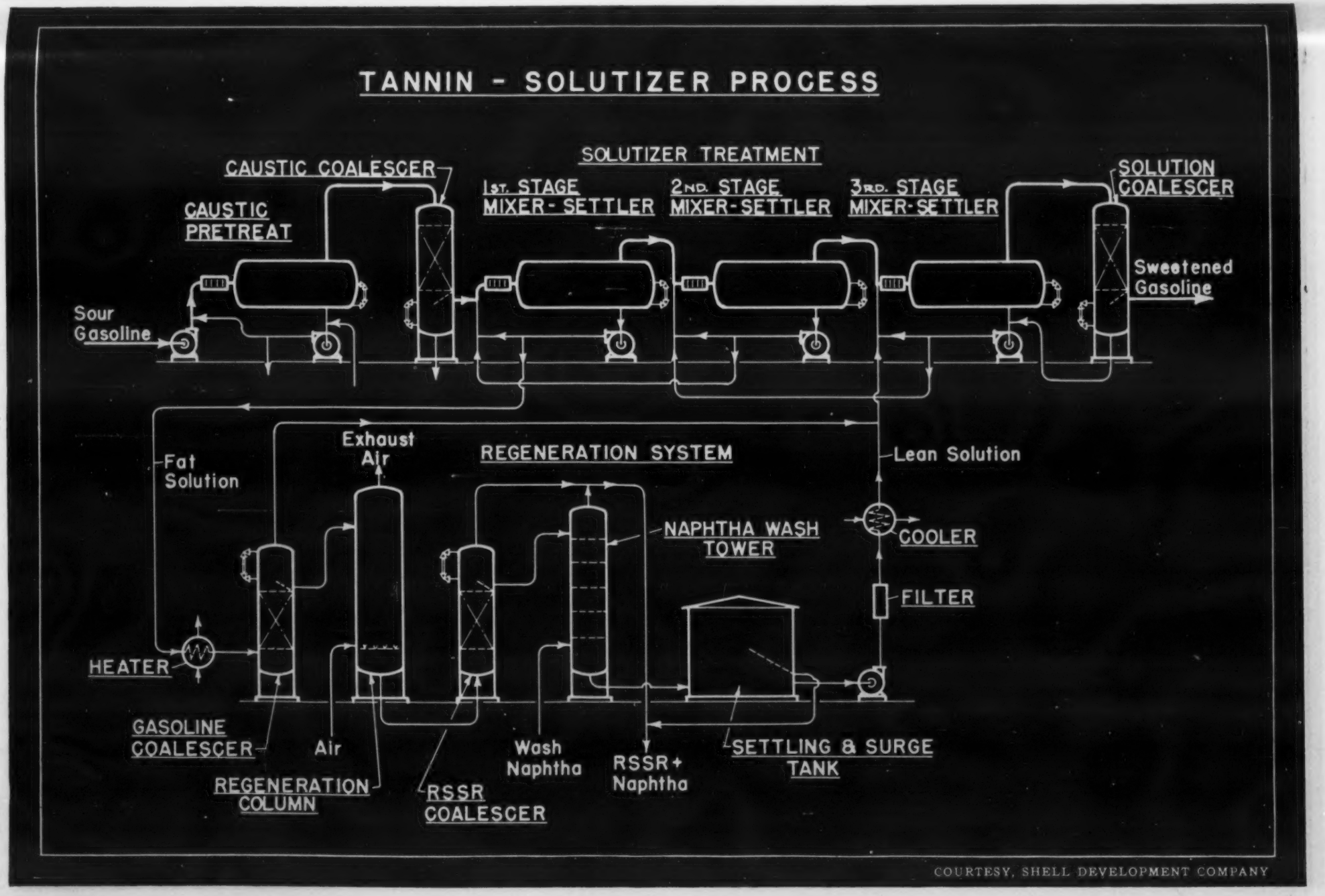
After pretreatment the sour hydrocarbon feed enters the extraction system in which it is countercurrently contacted with a suitable solutizer solution. The solution may contain added isobutyric acid solutizer, or naturally occurring alkyl phenols may be utilized for this purpose if their concentration is sufficient in the hydrocarbon fraction to be treated. The extraction equipment may be composed of mixer-settler stages, such as the typical three-stage system shown on the flow diagram, or other suitable contacting equipment such as towers packed with Raschig rings or of perforated tray design. The sweetened hydrocarbon flows from the extraction system to storage or subsequent operations.

Treating solution from the extraction step is preheated to a temperature of 110-130° F. and flows to the regeneration column in which the extracted mercaptans are catalytically oxidized to the corresponding disulfides by air blowing. This operation may be accomplished in a column devoid of packing equipped with efficient air dispersers or in any standard liquid-gas mixing device. The regenerated solution is coalesced for removal of entrained disulfide oil and the portion of this material remaining in solution is removed by a simple countercurrent or batch naphtha-wash step. The naphtha requirements for virtual elimination of disulfides from the lean treating solution are small, amounting in volume to only 1-2 percent of the hydrocarbon-treating rate, and may be disposed of in third-grade fuel or added to reforming or cracking stock for conversion of the disulfides to hydrogen sulfide. The lean solution then is cooled and returned to the extraction system, thus completing the cycle.

Additional equipment consists of coalescers appropriately located to safeguard against entrainment of hydrocarbon or solution and a filter to prevent accumulation of possible side products of oxidation and extraneous material in the treating solution.

This process easily achieves mercaptan removal to the 0.001-0.003 weight-percent mercaptan sulfur level when treating a wide range of straight-run or cracked stocks, either low-end-point or full-boiling-range gasolines, having a naphthenic or paraffinic crude source. Mercaptan reduction to the doctor-negative specification may be obtained in many cases by means of more stringent operating conditions and increased number of extraction stages. The process is characterized by a simple and flexible design which is particularly readily adaptable to utilization of existing equipment; and, due to the low operating temperatures and non-corrosive nature of the treating solution, no corrosion-resistant metals are required. Simplicity of operation results in low labor costs and chemical and utility consumption is nominal. The profitability gained from the saving in TEL resulting from the sulfur removal in most cases exceeds the low overall operating cost, so that an early payout of the original capital investment of an installation is realized.

The Tannin-Solutizer Process is licensed by Shell Development Company under patents of Socony-Vacuum Oil Company and Shell Development Company.


TANNIN - SOLUTIZER PROCESS
SOLUTIZER TREATMENT
CAUSTIC COALESCER
CAUSTIC PRETREAT
1ST. STAGE MIXER-SETTLER
2ND. STAGE MIXER-SETTLER
3RD. STAGE MIXER-SETTLER
SOLUTION COALESCER
Sour Gasoline
Sweetened Gasoline
Exhaust Air
REGENERATION SYSTEM
Fat Solution
Lean Solution
NAPHTHA WASH TOWER
COOLER
FILTER
HEATER
GASOLINE COALESCER
Air
Wash Naphtha
RSSR + Naphtha
SETTLING & SURGE TANK
REGENERATION COLUMN
RSSR COALESCER
COURTESY, SHELL DEVELOPMENT COMPANY

Thermal Delayed Coking Process

FOSTER WHEELER CORPORATION

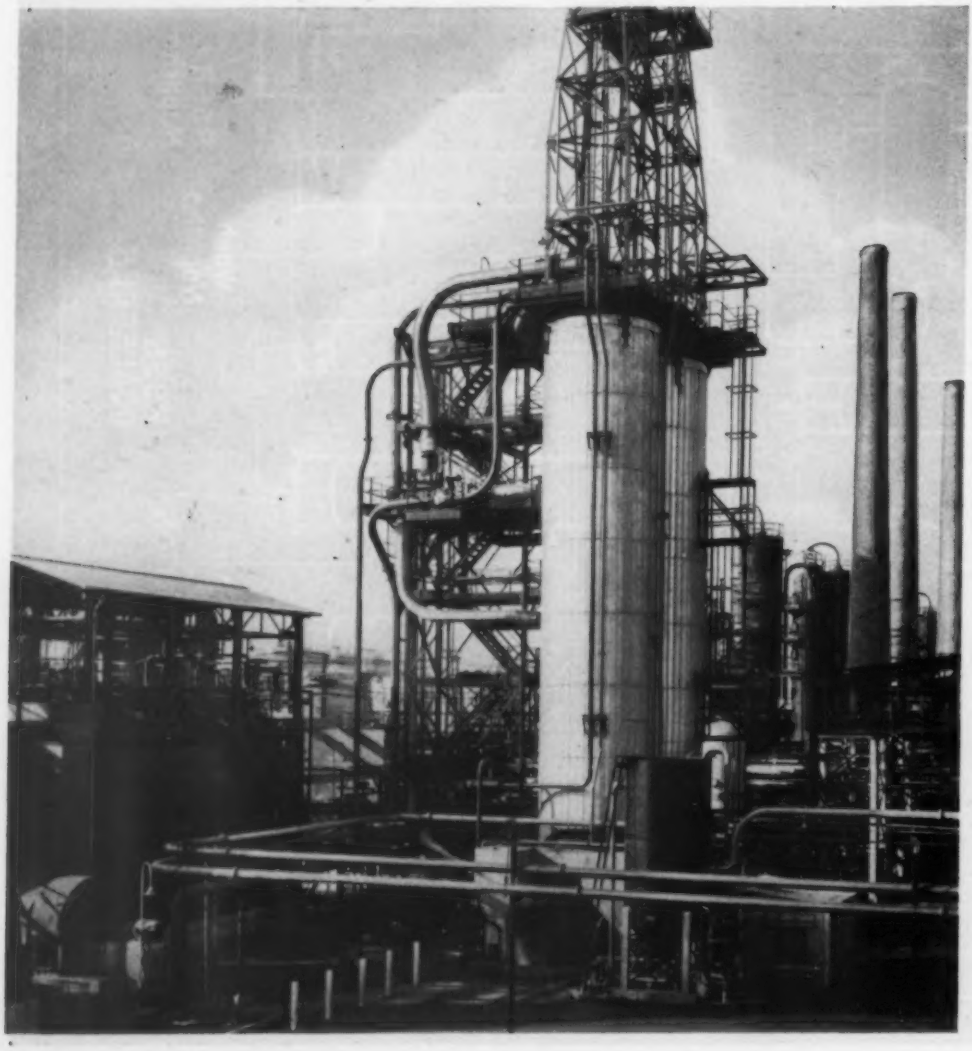
IN THE Thermal Delayed Coking Process stocks such as reduced crude, straight-run pitch or cracked tars are heated to a temperature sufficient to cause their heaviest components to turn to coke in a time chamber, from which the coke is intermittently removed.

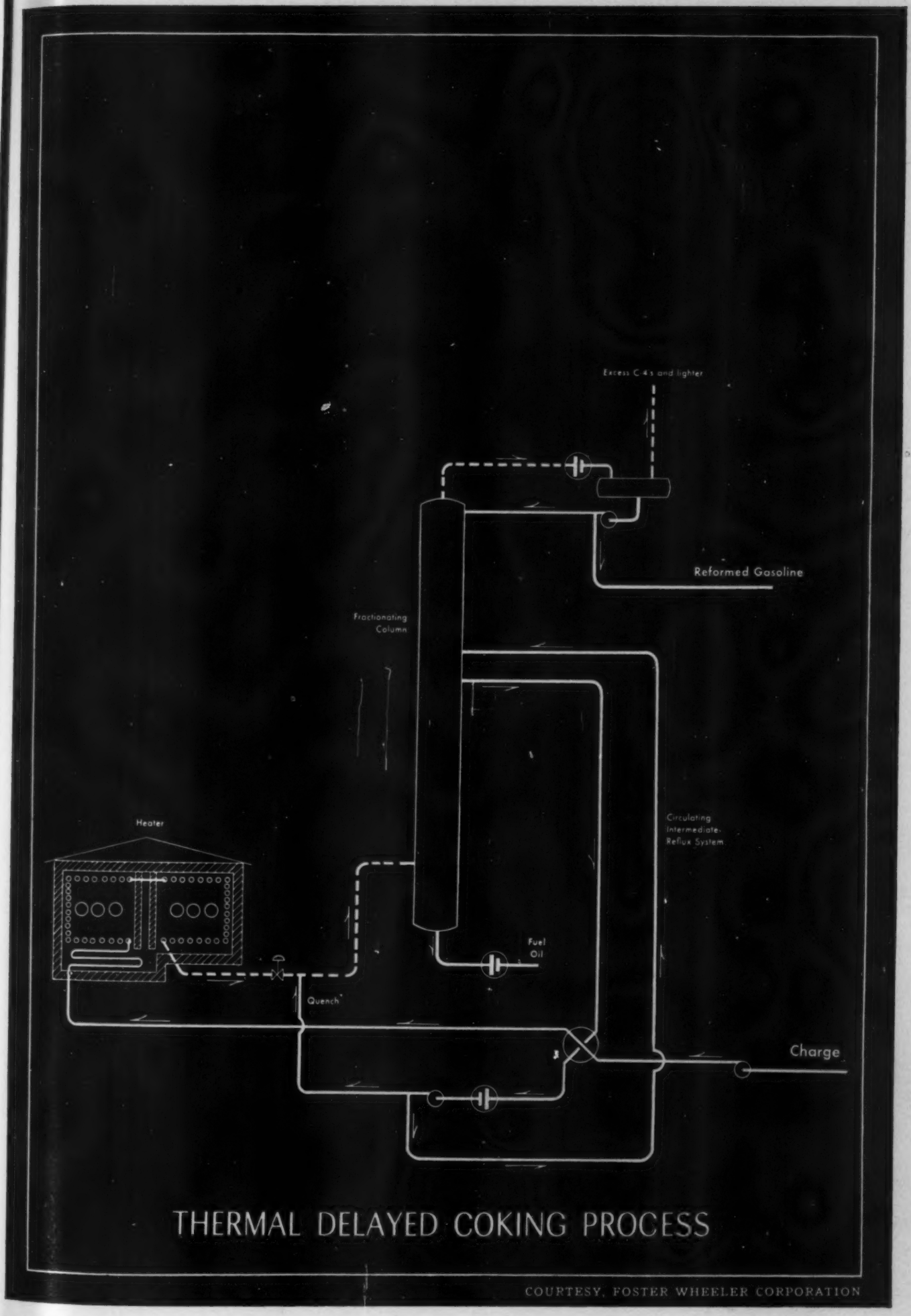
The primary objective of delayed coking is to produce, from heavy carbonaceous oils, clean gasoils which are suitable charges for thermal or catalytic cracking. When demand and market realization for coke are adequate, delayed coking of the heavy surplus oil stock can be a very profitable operation. A yield as high as 20 percent of 400° F. end-point gasoline from the delayed coking of reduced crude represents an added asset in the overall economic balance.

Thermal delayed coking units may be designed to operate as viscosity-breakers when there is either no demand for coke at a fair return, or when there is a surplus supply of heavy residues.

Two or more coking chambers usually are provided for the intermittent removel of coke. The "hydraulic method" is looked upon with much favor by operating men because it consumes the smallest amount of time and produces the smallest percentage of coke fines. In this method a vertical hole about 18 inches in diameter is bored by a high-pressure water jet through the coke bed after the coke has been prepared by adequate steaming. The next step is the cutting of the coke into layers by using high-pressure water jets applied by means of rotating nozzles on the end of a pipe extending down to the bottom of the vertical hole. This cutting works upward until the entire body of coke is removed from the chamber. The water used for cutting the coke also may be used to convey the coke in a conveying system from the coke chamber to the stock pile.

The charge, a heavy reduced crude or cracked tar, is pumped through the convection section of the "Downdraft" heater and enters the radiant section where it is heated to a temperature high enough to effect vaporization of all light and heavy gasoils contained in the charge. This liquid-vapor mixture enters the fractionating column and the unvaporized heavy fuel oil fraction of the charge is pumped from the bottom of this fractionating column into the other cell of the "Downdraft" heater where further vaporization takes place. This combination of heavy oil vapor and liquid residue (in which coke-forming substances are highly concentrated) enters one of the coking chambers where as a result of the combination of time and temperature, coke is formed. This coke accumulates until the chamber is filled and the stream is then diverted to the other coke chamber. The vapor leaving the coke chamber contains an appreciable quantity of low-boiling fractions, the result of cracking both in the heater coil and in the chamber itself. This vapor enters the fractionating column and is separated into gas and gasoline at the top of the column and a gasoil sidestream.





THERMAL DELAYED COKING PROCESS

COURTESY, FOSTER WHEELER CORPORATION

Thermal Reforming Process

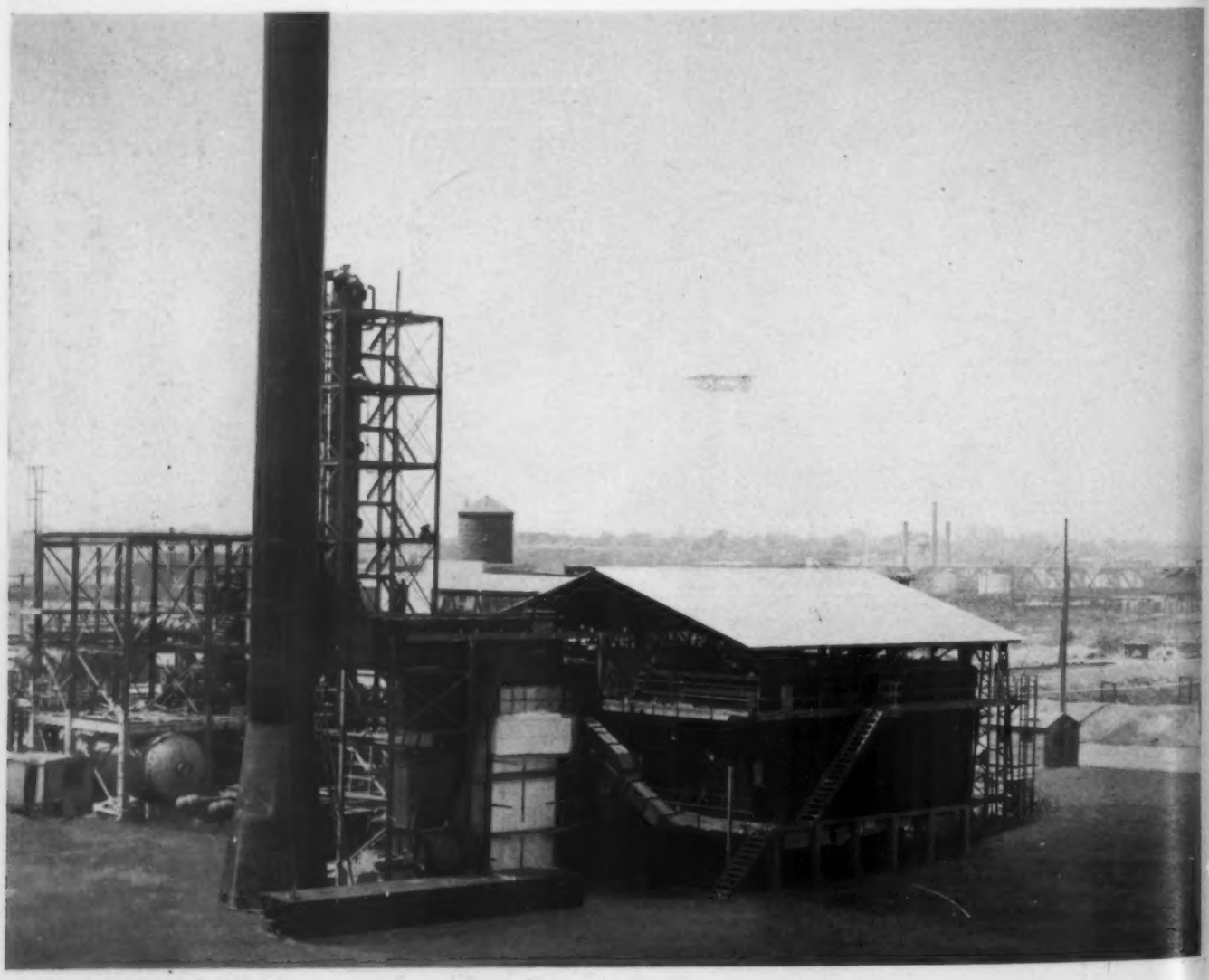
FOSTER WHEELER CORPORATION

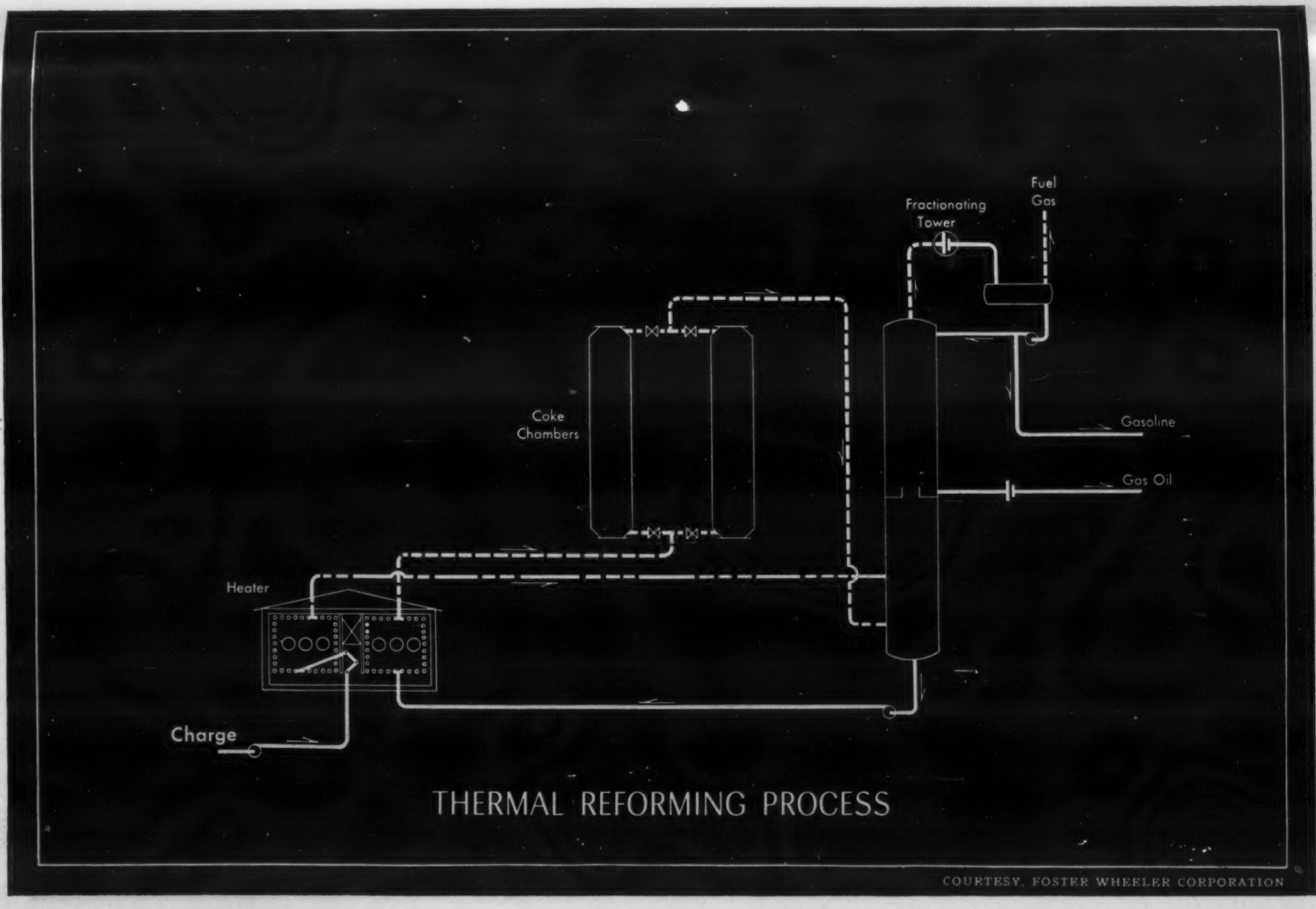
YIELD of 75 to 85 percent of 65 to 75 ASTM octane from a 45 to 50 ASTM octane charge represents the significant function of a thermal reformer in modern petroleum refining.

Most of the low-octane fractions in straight-run naphtha are the straight-chain paraffin hydrocarbons with boiling points between 250° F. and 400° F. When the crude available to a refiner contains a large percentage of these low-octane fractions, thermal reforming is utilized to convert them into higher-octane lower-boiling motor gasoline components.

Thermal reforming is widely practiced in the petroleum industry. Although the process is a very simple once-through operation, it is important that the equipment, particularly the furnace, be properly designed to operate satisfactorily under the severe conditions required.

The heavy low-octane reforming stock enters the direct-fired "Downdraft" heater after picking up part of the excess heat in the circulating quench oil and intermediate reflux. Reaction temperatures between 950° F. and 1050° F. are utilized depending upon the depth of the reforming required. Adequate soaking time is provided in the heater and the reformed gasoline then passes through a regulating valve, which maintains a pressure of 500 to 1000 psi on the heating and soaking coil, to the low-pressure fractionation part of the system. Before the reformed products enter the fractionator their temperature is quickly reduced by bringing them into contact with a comparatively low-temperature quenching oil for the purpose of reducing the formation of excessive coke. In the fractionator, a very small percentage of fuel oil residue is separated and passes out of the system to storage. The overhead combination of reformed gasoline and gas passes through a condenser to a receiver from which the reformed naphtha is pumped to a stabilizer. The gas, from the stabilizer, rich in C_3 and C_4 olefins, represents a very desirable charge for the catalytic polymerization unit.





THERMAL REFORMING PROCESS

COURTESY, FOSTER WHEELER CORPORATION

Thermofor Catalytic Cracking Process

HOUDRY PROCESS CORPORATION

THE Thermofor Catalytic Cracking (TCC) Process is of the moving-bed type and is licensed by Houdry Process Corporation.

As shown in the flow diagram the major parts of the catalytic section consist of reactor, kiln, and catalyst elevators. The reactor is a cylindrical vessel with catalyst and vapor inlets at the top and a catalyst-vapor disengager and oil-purge section at the bottom. Catalyst pellets flow downward by gravity, along with the hydrocarbon vapors, forming a solid moving bed. The depth of catalyst bed in the reactor is variable and controls the space velocity. After passing through the reaction zone the catalyst contains a small amount of carbonaceous deposit. Oil vapors and adsorbed oil are purged from the catalyst and surrounding voids in the purge zone after which the catalyst flows into the spent-catalyst elevator which operates at atmospheric pressure. This elevator transports the catalyst to the top of the kiln where it is discharged into a hopper feeding into the kiln. The catalyst flows downward by gravity through the kiln and the carbonaceous deposit is removed by zone regeneration with air at about 1.0 pound gauge pressure. Each burning zone has its own air supply and water-cooling coils, permitting a close control of catalyst temperature which thereby protects the catalyst from deactivation by excessive temperatures.

The kiln is designed to effect substantially complete removal of carbon from the catalyst by providing the required number of zones and residence time in each zone. Regenerated catalysts from commercial kilns normally average 0.2 weight-percent carbon or less. Most of the heat of combustion is converted into high-pressure steam by circulating water through the cooling coils and flashing in a separate steam drum.

The regenerated catalyst is returned from the bottom of the kiln by a second elevator to the reactor hopper, completing the catalyst cycle. On units charging less than 7000 barrels per day, a single elevator having partitioned buckets carries both spent and regenerated catalyst streams. This type of elevator is shown on the flow diagram.

It should be noted that the elevators operate at atmospheric pressure, providing a positive separation of the kiln and reactor and assuring that a momentary upset on either kiln or reactor will not force a shutdown of the unit.

Catalyst within the TCC unit is maintained at maximum particle size by continuously by-passing a small portion of the regenerated catalyst stream through an elutriator where fines are separated with countercurrent air flow. Whole catalyst pellets from the elutriator then flow by gravity to the regenerated catalyst elevator.

An inert-gas seal is used to prevent the loss of hydrocarbon vapors out the top of the reactor. As shown, flue gas from the kiln or from the flue-gas generator is introduced at the bottom of a catalyst seal leg located between the feed hopper and reactor. The length of this seal leg is sufficient to provide a pressure drop exceeding the reactor pressure. The main part of the seal gas is vented to the atmosphere from the feed hopper, with a small portion passing through the reactor.

Aside from the catalytic equipment described above, heat exchange, fractionation, etc., are conventional.

The scheme of charge preparation shown in the flow diagram consists of heating reduced crude and flashing at approximately 15 psig pressure. The flashed distillate comprises the TCC charge. If desired the flashed residue may be vacuum-distilled or propane-deasphalted to obtain additional charge. In this situation the recovered heavy gasoil is heated to 700-800° F. and injected as a liquid at the top of the reactor.

The following table presents typical yields from a commercial plant using synthetic catalyst and cracking a coastal naphthenic charge stock in an essentially single-pass operation:

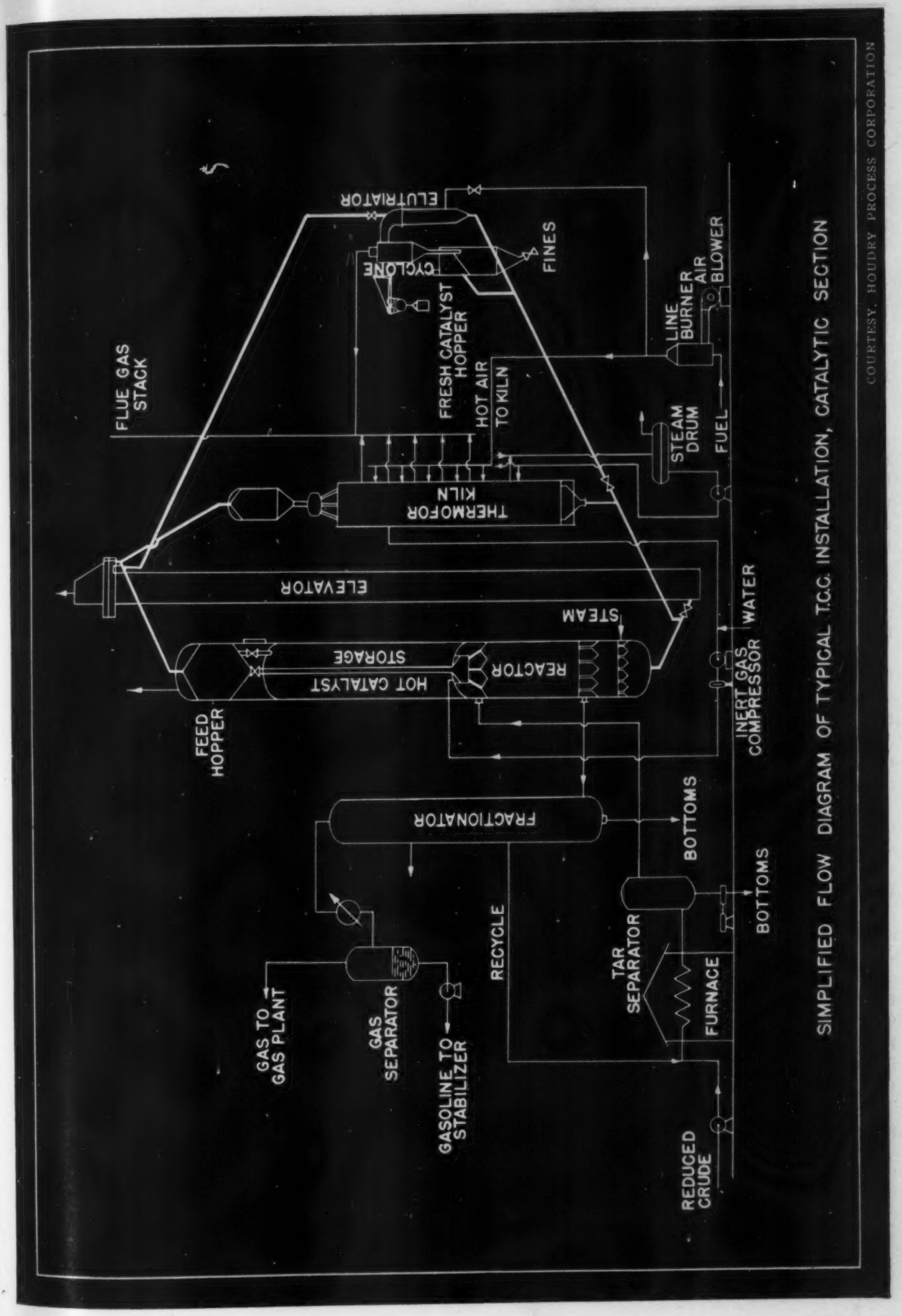
Cracking Coastal Naphthenic Gasoil for Motor Gasoline

	Vol. Percent	°API	Aniline Point °F.	DISTILLATION 10 Percent	DISTILLATION 50 Percent	DISTILLATION 90 Percent	OCTANES CFR-M Clear	OCTANES CFR-M 3 cc TEL	OCTANES CFR-R Clear	OCTANES CFR-R 3 cc TEL	Percent S
Charge	100	28.1	136	472	528	624	...	...	...	...	0.14
Products:											
10lb R.V.P. TCC Motor-Gasoline*	53.9	53.3	...	120	240	374	83.5	90.3	94.2	100.0	0.019
Excess C_4 Fraction	12.1	...	...	...	...	...	...	...	...	...	...
Catalytic Gasoil	34.0	21.7	88	470	510	600	...	...	...	...	0.07
Liquid Recovery (C_4+)	100.0	...	...	...	...	...	...	...	...	...	...
Dry Gas, Weight Percent	7.6	...	...	...	...	...	...	...	...	...	...
Coke, Weight Percent	2.1	...	...	...	...	...	...	...	...	...	...

SUMMARY OF OPERATING CONDITIONS

Total Space Velocity V./Hr./V.**	0.75	Vapor Inlet Temp. °F.	924
Catalyst	Bead	Catalyst Inlet Temp. °F.	950
Catalyst Activity Index	36	Avg. Reactor Pressure, psig	5.0
Process Steam	None		
Type Flow	Concurrent		

* Cu Dish Gum—6 mg; ASTM Gum—3 mg; Oxygen Bomb Stability 20 + Hrs.
** Contains 5.3 vol. percent of internal recycle.


ELUTRIATOR
CYCLONE
FINES
FRESH CATALYST HOPPER
HOT AIR TO KILN
LINE BURNER
AIR BLOWER
FLUE GAS STACK
THERMOFOR KILN
STEAM DRUM
FUEL
ELEVATOR
STEAM
HOT CATALYST STORAGE
REACTOR
WATER
INERT GAS COMPRESSOR
FEED HOPPER
FRACTIONATOR
BOTTOMS
BOTTOMS
RECYCLE
TAR SEPARATOR
FURNACE
GAS TO GAS PLANT
GAS SEPARATOR
GASOLINE TO STABILIZER
REDUCED CRUDE
SIMPLIFIED FLOW DIAGRAM OF TYPICAL T.C.C. INSTALLATION, CATALYTIC SECTION
COURTESY, HOUDRY PROCESS CORPORATION

Three-Stage Distillation Process

E. B. BADGER & SONS COMPANY

CRUDES usually are distilled under substantially atmospheric pressure for the removal of straight-run products through kerosine and light gasoil, and the reduced crude is then subjected to further distillation under vacuum for the removal of lubricating-oil fractions and the production of a heavy residuum or asphalt. These operations may on occasion be preceded by a flashing operation under mild pressure to recover light gasoline and to condition the crude for further processing.

The flow diagram on the opposite page illustrates a three-stage distillation unit designed and constructed by E. B. Badger & Sons Company, similar in general concept to the majority of modern Badger-built crude distillation units, and incorporating these three operations. Three-stage crude distillation when applied to light and medium crudes offers increased flexibility of operation; a means of combatting corrosion due to sulfur, salt and water, thereby resulting in greater on-stream efficiency; maximum yield of specification products; and improved control of fractionation.

In the first column, which usually operates at 25 to 75 psig, light gasoline is recovered overhead, and water and hydrogen sulfide are eliminated from the flashed crude. The advantages obtained in removing a light-gasoline cut in this manner before undertaking the fractionation of the intermediate cuts are as follows:

The wet crude charge is preheated in tubular exchangers at a low temperature level, minimizing corrosion difficulties from salt, water, and hydrogen sulfide. It is effectively dehydrated by flashing and stripping in the primary tower. This stripping is accomplished by reboiling the primary tower bottoms in a fired heater. Since no steam is employed for stripping, very-low-boiling products can be recovered overhead without danger of condensing water in the tower, thus minimizing corrosion from this source. Controlled amounts of light hydrocarbons can be kept in solution in the light overhead cut by operating at suitable pressures, whereas, it is impracticable to do this in the main tower at atmospheric pressure. The control of separation between the gas and light gasoline and the light naphtha can be accomplished separately and therefore more effectively. Furthermore, a properly conditioned and dried oil is obtained for charging to the fired heater, thus overcoming difficulties due to salt deposition in the heater tubes. Additionally, the top section and condensing equipment in the atmospheric tower can be designed to take advantage of the elimination of the corrosive agents in the primary tower.

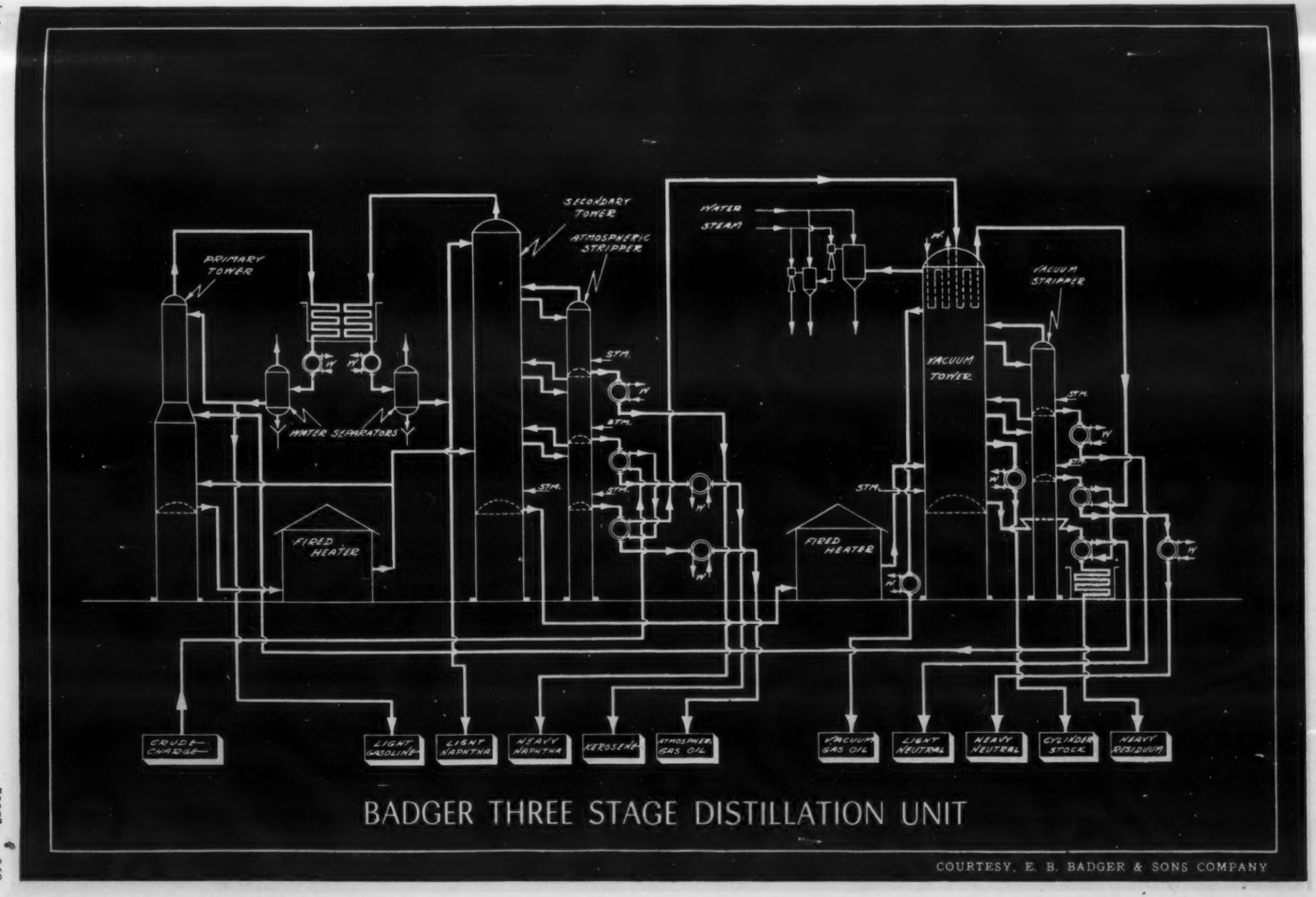
The secondary tower operates at substantially atmospheric pressure and is designed to remove light naphtha overhead, a heavy naphtha fraction and kerosine and gasoil as side streams. Steam is used for stripping the side streams and bottoms, but since the light gasoline is removed in the primary tower, the temperature level is sufficiently high to prevent condensation of water on the upper trays. The heat requirements for the atmospheric tower are provided by the same heater used for reboiling the primary tower bottoms.

In larger units or in special cases it becomes economical to provide two fired heaters, instead of one as shown in the diagram, for the first two stages. One heater circulates primary bottoms for reboiling and the other serves as a preheater for the secondary tower. Condensers can be the submerged-coil type as indicated for this unit or multitubular or direct-contact type according to conditions.

The vacuum tower produces a heavy gasoil overhead, light and heavy neutral and cylinder stock as side streams, and a heavy residuum as a bottoms product. As indicated on the flow diagram, the vacuum condensers have been mounted in the top of the tower although they may be supported above and outside the tower. In smaller units where the vapor lines are of moderate size, the condensers may be placed at a lower level for convenience. Pumps generally are used for removing vacuum distillates from the system, but in some instances where pumps are subject to severe corrosion such as by naphthenic acids in the distillate, steam lifts and barometric legs are used.

The character of the heat-exchange system is dictated by the nature of the stocks handled, and economical heat recovery varies greatly with each unit. Alternate paths of flow usually are provided for different operations.

Badger crude distillation units are designed for ease and smoothness of operation, minimum product loss while changing charge stocks, high stream efficiencies, and minimum down-time during unit turnarounds.



BADGER THREE STAGE DISTILLATION UNIT

COURTESY, E. B. BADGER & SONS COMPANY

Three-Stage Distillation Process

FOSTER WHEELER CORPORATION

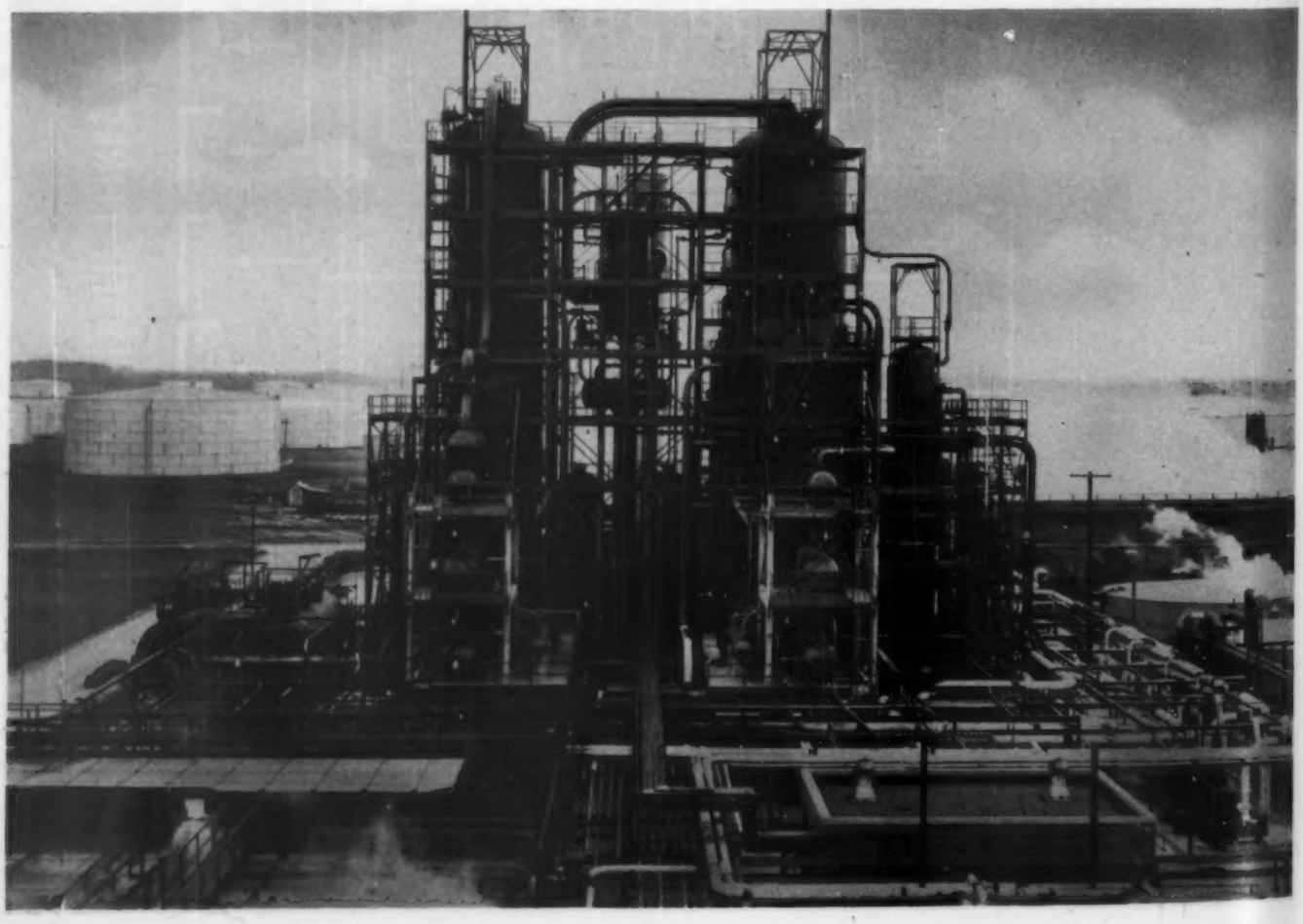
PREFABRICATION and the separation of naphthas, refined oils, and light gasoils in an atmospheric column, plus the removal of heavy gas oils and lubricating distillates from the reduced crude in a vacuum column is the fractionation sequence in the three-stage crude oil distillation process.

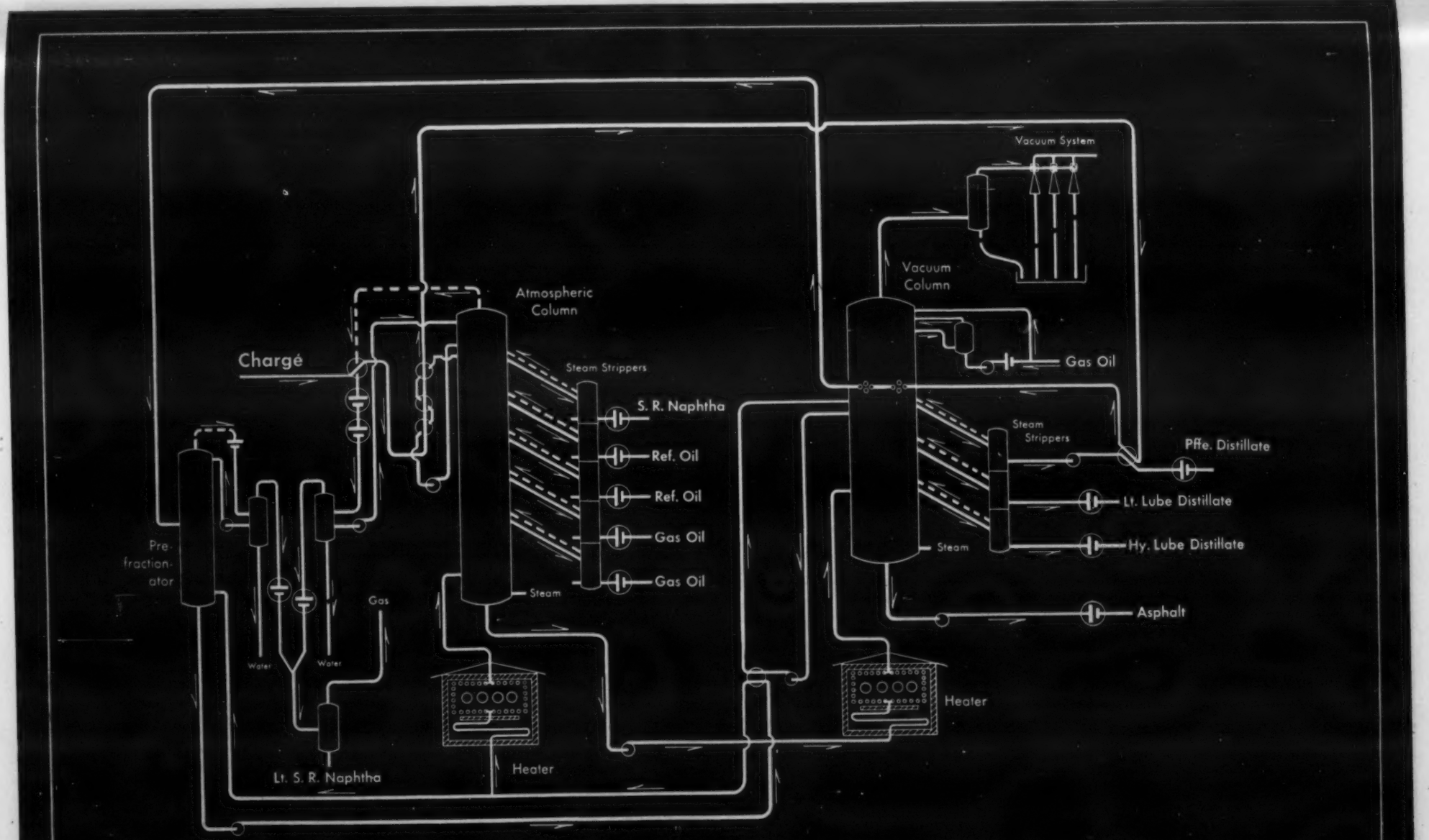
Prefractionation flashes-off from the crude, hydrogen sulphide, and water, thereby concentrating the low-temperature sulphur-corrosion problem to one small section of the unit. It also removes the lighter fractions from the crude, thereby increasing top-of-tower temperature in the atmospheric column and avoids steam condensation therein. The heat required for the prefractionator is obtained almost entirely by heat exchange.

The atmospheric distillation part of the system removes from the crude, at normal temperature, the remaining naphtha, the refined oil, and light gasoil. Further atmospheric distillation of the crude would be attended with a certain degree of thermal degradation which is not conducive to the production of the highest quality of lubricating distillate; therefore, the reduced crude from the atmospheric column is further reduced in the vacuum distillation part of the system to produce the maximum yields of the various grades of lubricating stocks available in the particular crude charge to the unit.

The crude enters the unit through a vapor heat exchanger, then through a series of exchangers where it absorbs heat from a circulating intermediate reflux stream from the atmospheric fractionating column. Further temperature increase is obtained by heat exchange with the paraffin distillate stream from the vacuum column; the crude stream then passes through tube bundles located in the vacuum tower. The crude stream, so preheated, then enters the prefractionating tower. After flashing off hydrogen sulphide, water and a light naphtha fraction the partially reduced crude from the prefractionator is pumped through a heat exchanger where it absorbs heat from a circulating intermedate reflux of the vacuum tower; then passing to the "Downdraft" heater where it is raised in temperature to effect vaporization of the remaining naphtha, the refined oils and light gasoils.

The reduced crude from the atmospheric tower is pumped directly to another "Downdraft" heater where it absorbs sufficient heat to effect vaporization of the remaining gasoils and the light and heavy lubricating oil stocks, which are then fractionally separated in the vacuum tower.





THREE STAGE CRUDE DISTILLATION PROCESS

COURTESY, FOSTER WHEELER CORPORATION

Three-Stage Distillation Process

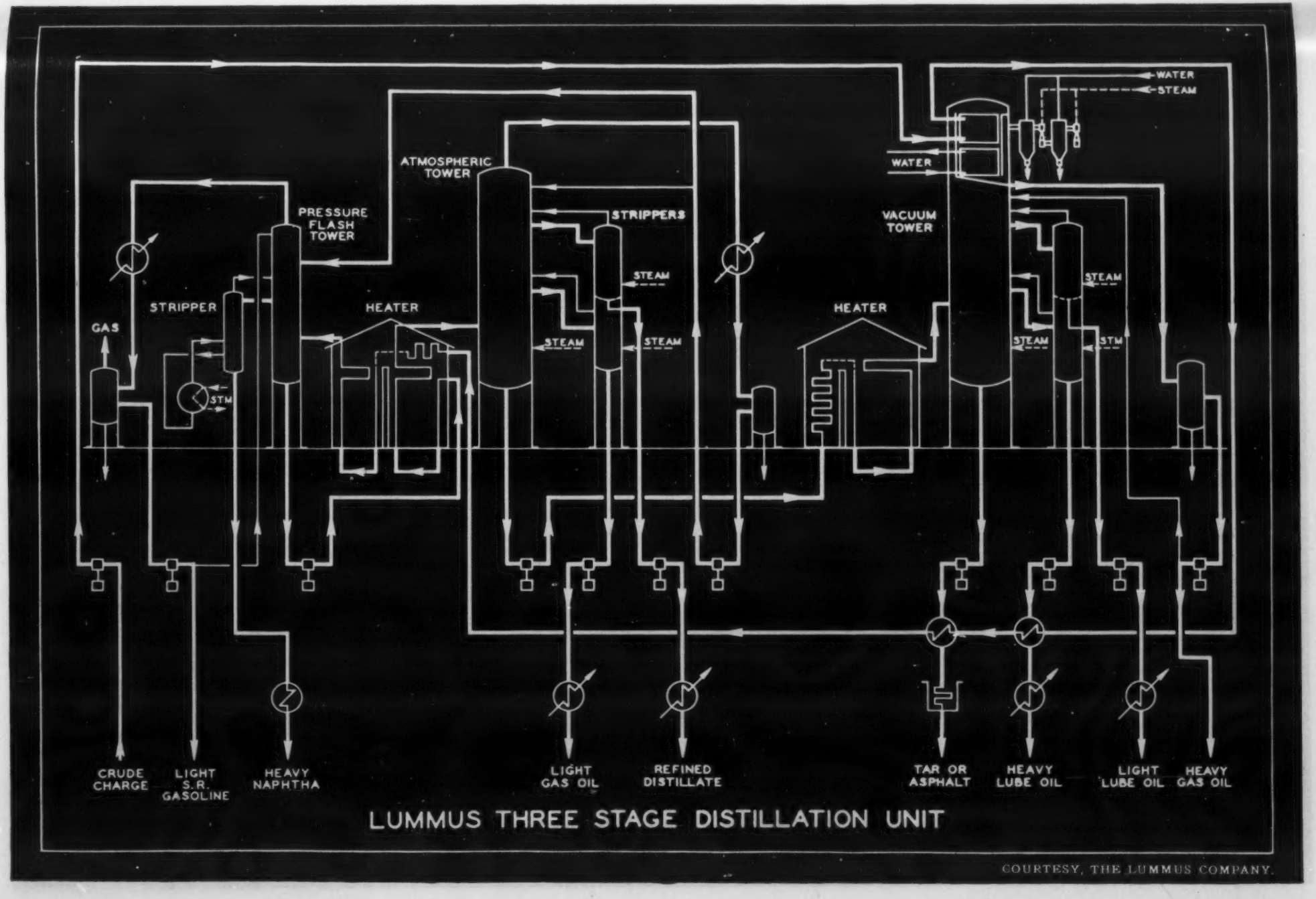
THE LUMMUS COMPANY

LUMMUS three-stage distillation units (see flow sheet opposite) are employed for processing of light sour crudes with the first stage operating without steam at 40-50 psig pressure to facilitate condensation of the light hydrocarbons but at the same time avoid condensation of acidic moisture in the tower. In this tower the light gasoline is distilled overhead and heavy naphtha is withdrawn as a side stream. The second stage operates at substantially atmospheric pressure and the third stage operates under vacuum.

The crude oil passes through heat exchangers to the convection bank of the oil heater and from there to the primary pressure tower. One radiant section of the heater is used for reboiling the primary flash tower. The side stream of heavy naphtha also is provided with a reboiler, the heat for this being provided by a separate hot stream from one of the other towers. Several marked advantages are obtained by operating the primary tower under pressure. Since the heat added to the crude oil is at a fairly low temperature level, there is less difficulty from corrosion due to sulphur, salt and water in the crude. Difficulties from corrosion also are reduced by eliminating stripping steam, since the water content of the crude normally is not sufficient to cause condensation of water on the decks of the column. Condensation of the overhead light gasoline is carried out in two stages. The primary condensate is used as reflux, the excess passing through a subcooler as net product and being used to absorb gas remaining uncondensed in the primary condenser. Using this system it is possible to obtain a product with high vapor pressure and low end point and to operate on "wild" crude with minimum production of gas. This product can be pumped directly to the stabilizer, and since it contains most of the gas which is normally not condensed no compressor is needed and there is no additional load on the refinery gas-recovery plant.

The secondary tower, operating at substantially atmospheric pressure, is designed to remove kerosine and gasoil fractions. In order to have complete control of the initial boiling point of kerosine, this fraction is removed as a side stream. The overhead from this tower thus represents a fraction intermediate between heavy naphtha and kerosine. Most of this material is used as a reflux to the atmospheric tower, while a small percentage is returned to the primary tower as intermediate reflux. Steam is used for stripping of both side streams and bottoms but since the temperature level at which this tower operates is fairly high, there is little difficulty with corrosion because of the fact that no condensation of water takes place in the tower. The heat requirements for the atmospheric tower are provided by a second radiant chamber of the heater. Each of these chambers is fired independently, so that firing is under absolute control.

The third tower of the system is operated under a vacuum for the production of lubricating-oil distillates. The overhead stream on this tower is a heavy gasoil. The various lubricating-oil fractions are removed as side streams while the bottoms is heavy tar or asphalt, depending upon the type of crude oil being processed. Steam is employed for separating both the side streams and the residue. In order to avoid the effect of high temperatures on the quality of lube-oil distillates produced, the oil heater is designed so that vaporization takes place within the heater itself. The vacuum fractionating tower design includes the Lummus vacuum condensing system which features the installation of these units in the top section of the tower itself rather than on an external structure.



LUMMUS THREE STAGE DISTILLATION UNIT

COURTESY, THE LUMMUS COMPANY.

Tretolite Desalting Process

TRETOLITE COMPANY

WITH the introduction of modern refining facilities, the presence of inorganic salts in crude oil was found to be an obstacle to trouble-free operations. Equipment corrosion and plugging, as caused by these salts, resulted in frequent shutdowns and high maintenance costs.

Recognizing the fact that most salts were carried in the oil as residual emulsified brine particles, many refineries resorted to the use of settling drums on the crude units to separate these emulsion particles. Partial salt removal was effected in some cases; however, in all cases, removal was insufficient to permit smooth operations.

In 1928 Tretolite Desalting was introduced to improve the degree of salt removal. On-stream time was extended from a matter of days to a matter of weeks on the first applications, but the nature of the problem dictated the need of salt removals in the 90 percent range.

The present Tretolite Desalting Process was designed to this end. The process is simple and economical, and can be utilized in the continuous operation of the crude stream to the flash tower or stills. A minimum of equipment is required, and, in places where settling drums already are installed, only minor modifications are required to convert the unit to the process.

The process consists of injecting a small amount of chemical destabilizer into the crude oil and contacting the oil with fresh water to wash out the residual brine and salt particles. The water then is permitted to coalesce and separate in a settling drum, carrying off the salt and some other detrimental materials. The chemical reagent serves as both a demulsifier of the residual emulsion in the crude, as well as a destabilizer for the new emulsions formed by the addition of the fresh water. This action permits better contact of the wash water with the brine particles. The presence of the destabilizer further aids in the coalescing of the dispersed water particles, giving a quick water separation and a clean interface in the settling drum.

Advantage is taken of the regular refinery equipment, such as charge pump, heat exchangers, and piping, to obtain the dispersion of chemical reagent and the fresh water phases; therefore, mixing valves are only necessary where a high degree of contact is required. A mixing valve usually is installed, but the pressure drop across the valve is held to a minimum.

The chemical requirement is in the range of 1 to 2 gallons per 1000 barrels of crude, depending upon the characteristics of the crude. The fresh water requirement is in the range of 3 to 7 percent of the crude stream, depending upon the nature and amount of the salts present. Operations are carried on in the crude unit at whatever temperature is available from the regular heat exchanger equipment. The settling drum is placed in the system at the point where sufficient pressure is still available to prevent vaporization of both the crude and water phases. Operating temperatures vary in the range of 180° to 300° F. The higher temperatures are best adaptable to the more viscous crudes, and, in most cases, the higher the temperature, the more efficient the desalting operations.

Settling-drum size is determined by both the rate of through-put and the nature of the crude. Average oil retention time within the drum is about 30 minutes, with a water level maintained at the proper height to insure a thorough washing of the crude. The water level is controlled by an automatic instrument. A back-pressure regulator is used on the crude outlet to maintain uniform back pressure upon the system.

This process is being applied at refineries throughout the country, and on all types of crude oils. Installations range from 1,000 to 35,000 barrels per day per unit. Salt removals range from 90 to 98 percent, with the water carry-over from the desalter equal to or less than the original residual emulsion content. The process has the added advantage of also removing a proportional percentage of insoluble solids, such as sand, shale, etc., which separate in the water phase of the settling drum. A partial removal of these materials reduces the amount of deposit that eventually occurs in the balance of the equipment.

The Tretolite Desalting Process is characterized by use of highly selective reagents intended specifically for such use, and described in various United States patents.* The Tretolite Company, 937 Pacific Avenue, Webster Groves, Missouri, is licensor.

* U. S. Patents 2,252,959, 2,310,673. Others pending.

TRETOLITE DESALTING PROCESS
CRUDE SUPPLY
TRETOLITE PUMP
RFC
CRUDE PUMP
FRESH WATER
RFC
WATER PUMP
BACK PRESSURE REGULATOR
DESALTED CRUDE
INTERFACE CONTROL
SETTLING DRUM
EFFLUENT WATER
MIXING VALVE
HEAT EXCHANGERS
COALESCING SECTION
COURTESY, TRETOLITE COMPANY

Tulsa Type Absorption Process

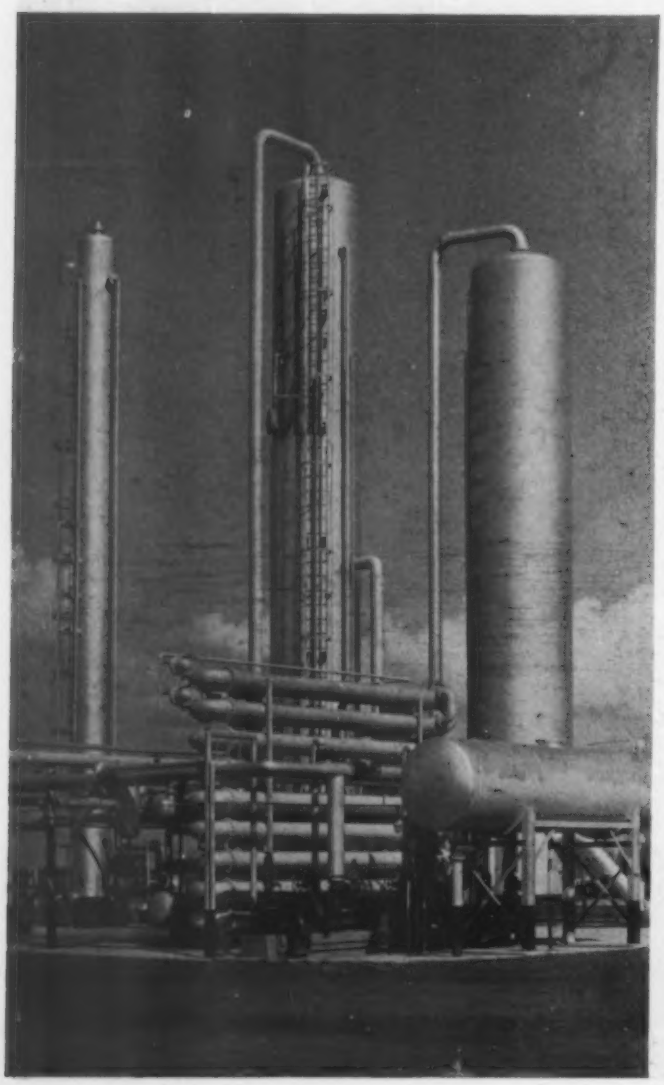
TULSA BOILER AND MACHINERY COMPANY

TULSA Boiler and Machinery Company, Tulsa, Oklahoma, present on the opposite page a typical flow diagram of its Tulsa Type Absorption Plant.

The inlet scrubber is equipped with a single mist extractor and both the absorber and the residue scrubber have double mist extractors to prevent entrainment of mineral seal oil into the residue gas line.

The oil-heat exchangers shown on the diagram are of the multitubular, fixed-tube-sheet design and this type is usually furnsihed; however, when it is necessary to clean both through and around the tubes, floating-head removable-tube-bundle exchangers are installed.

The evaporator-dephlegmator shown is of the combined type; however, in some instances, the evaporator and the dephlegmator are furnished as separate towers. The evaporator is furnished with two internal heating elements, one above each of the two lower trays, in which live steam is used as a heating medium through the tubes. This is a patented feature, and reduces the amount of steam required for heating the oil as there is no radiation loss. The oil passes around the tubes and inside the shells of the internal heating elements.



A small stream of hot mineral seal oil is taken off the bottom of the evaporator, heated with steam and introduced into the oil reclaimer. The oil vapors are condensed and the oil goes into the mineral seal oil storage tank. By continuously reclaiming the oil in this manner, the absorption oil remains in a clean condition.

The vapors from the raw make accumulator tank are taken into the recompressor or reabsorber for further processing.

In the fractionator hook-up on the diagram, a depropanizer, debutanizer, butane splitter and a deisopentanizer are shown. In some plants this would be more fractionators than necessary. In some installations, with the absorber working under high pressures, it would be advisable to install a deethanizer ahead of the depropanizer in the hook-up.

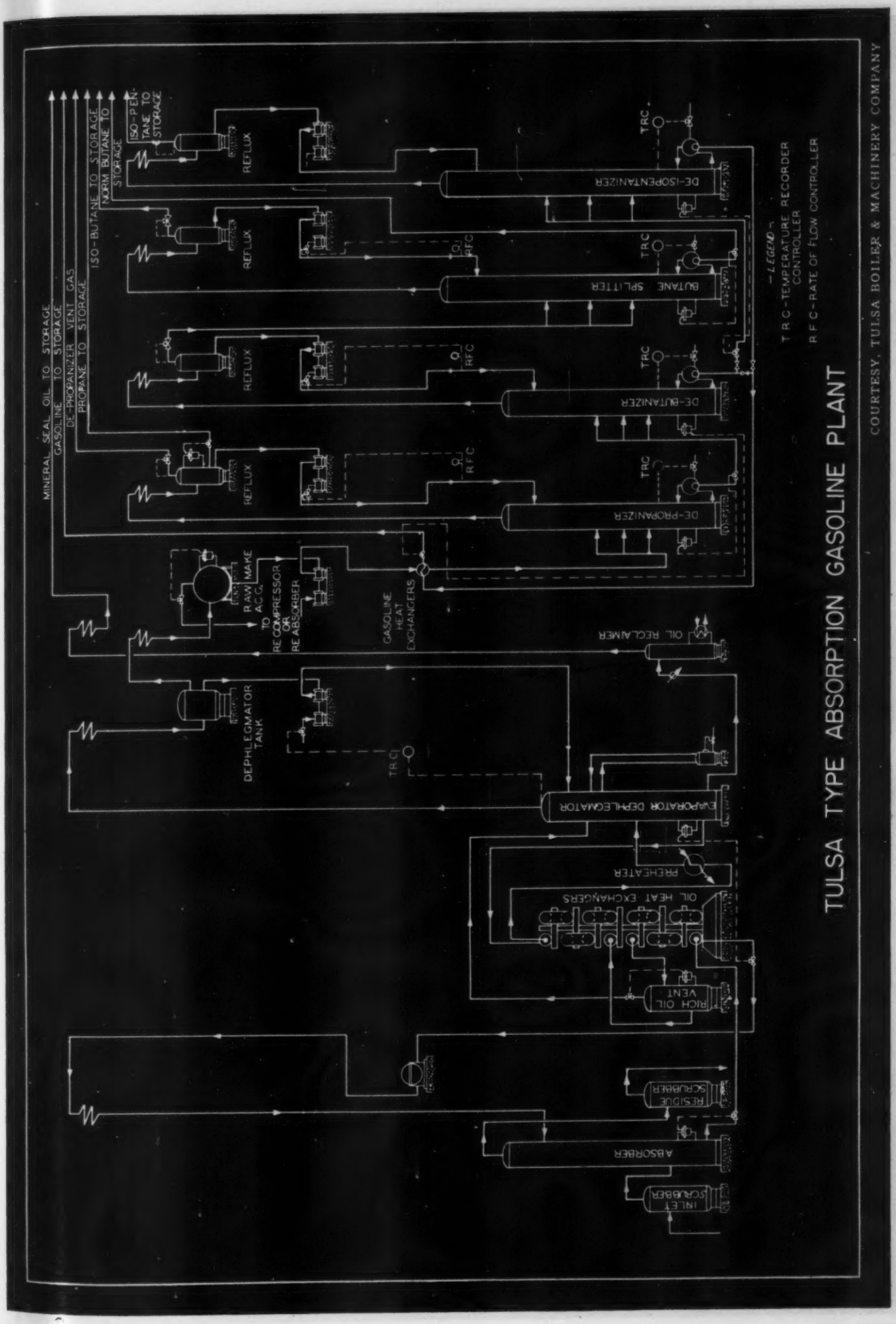
The coolers usually are of the atmospheric type with cast-iron or steel heads and inhibited Admiralty tubes. Some installations may require closed type, floating-head removable-tube-bundle coolers. The "Tulsa Type" atmospheric coolers have straight tubes. Both tube sheets are free to move back and forth to take care of expansion and contraction of the tubes. Scale from the tubes is shed by temperature shock which causes elongation of the tubes. By the tubes being taut and straight, dripping of water on the under side of the tubes is uniform, thus the coolers below the top cooler in each bank receive evenly distributed cooling water. The oil coolers, gasoline condensers, fractionator reflux condensers and fractionator final gasoline cooler are of this type, being built for the pressures necessary.

Gas-lift water wells at the side of the cooling tower, with gas jetting water over the tower are usual.

An absorber usually has 20 bubble trays for relatively high butane extraction. If higher butane extraction or propane extraction are desired, as many as 30 bubble trays are used. On some installations chilling elements are used in the absorber. This is also a patented feature. A refrigerant is introduced through the tubes of the chilling elements and the lean oil passes around the tubes and inside the shells of these internal chilling elements. The bubble trays in the fractionators number from 24 to 50, depending upon the service and degree of fractionation desired.

A gasoline heat exchanger is used on the feed line to the first fractionator for exchanging heat between the feed and the finished gasoline.

Rate-of-flow controls are used to control the amount of reflux to the various fractionators. Controls, regulators, and recording instruments are used throughout the plant to make it automatic in operation. The accompanying flow diagram is typical and could be changed as conditions warrant.


TULSA TYPE ABSORPTION GASOLINE PLANT
COURTESY, TULSA BOILER & MACHINERY COMPANY
MINERAL SEAL OIL TO STORAGE
GASOLINE TO STORAGE
DE-PROPANIZER VENT GAS
PROPANE TO STORAGE
ISO-BUTANE TO STORAGE
NORM BUTANE TO STORAGE
ISO-PENTANE TO STORAGE
REFLUX
DE-ISOPENTANIZER
BUTANE SPLITTER
DE-BUTANIZER
DE-PROPANIZER
TRC
RFC
LEGEND
TRC-TEMPERATURE RECORDER CONTROLLER
RFC-RATE OF FLOW CONTROLLER
OIL RECLAIMER
RAW MAKE
A.C.G.
TO RECOMPRESSOR OR RE ABSORBER
GASOLINE HEAT EXCHANGERS
DEPHLEGMATOR TANK
EVAPORATOR DEPHLEGMATOR
PREHEATER
OIL HEAT EXCHANGERS
RICH OIL VENT
RESIDUE SCRUBBER
ABSORBER
INLET SCRUBBER

Two-Stage Distillation Process

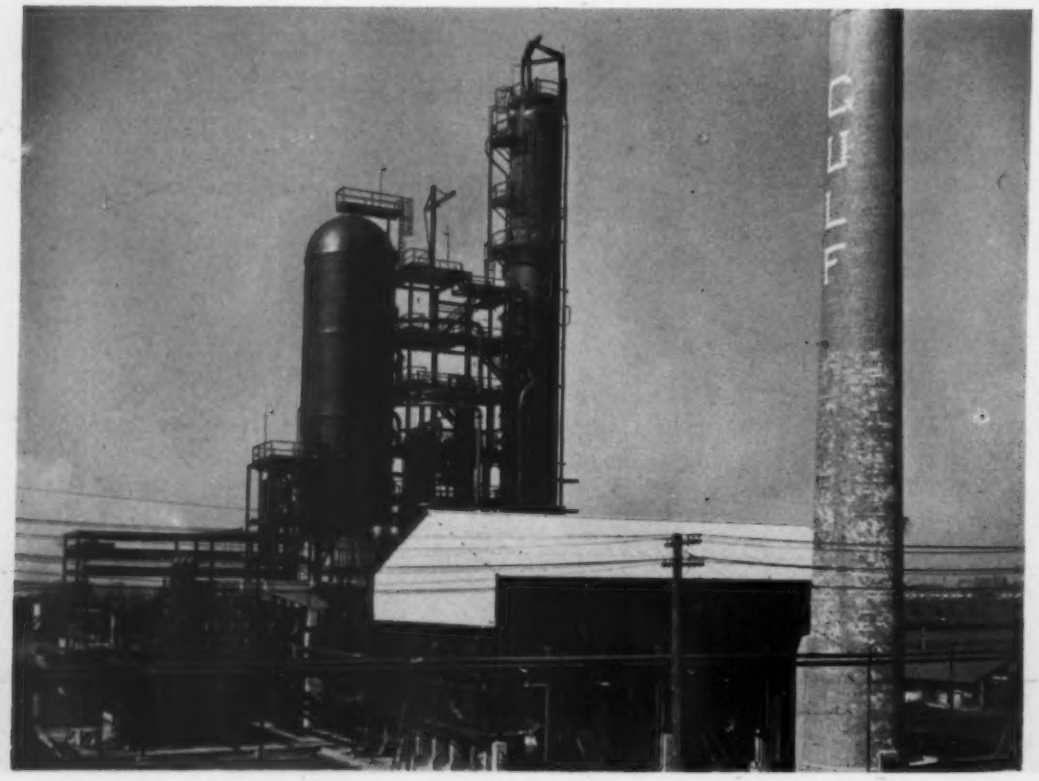
THE LUMMUS COMPANY

LUMMUS two-stage distillation units for the production of light gasoline, close-cut solvent naphthas, kerosine, cracking stock and fuel oil are in wide use and have excellent performance records. In these units (see flow sheet opposite) the two stages of distillation are both carried out at substantially atmospheric pressure with the temperature in the first stage being low enough to avoid the discoloration of kerosine. The temperature of the second stage is maintained at the lower limit of the cracking range to facilitate production of maximum yield of cracking stock.

The primary flashing of crude is accomplished through heat recovered by heat exchangers plus heat supplied by a coil including the convection bank and one radiant bank of a double-chamber Lummus oil heater. The products removed in the first stage include rubber solvent, varnish makers' and painters' naphtha, Stoddard solvent and kerosine. In order to have good operating control of the initial boiling point of rubber solvent, this product is condensed hot and partially weathered. The separation between rubber solvent and V.M.&P. naphtha can be easily obtained by efficient fractionation and without the use of any appreciable quantity of stripping steam. The separation between V.M.&P. naphtha and Stoddard solvent requires better fractionation than the foregoing separation, and since this latter is between the first and second side streams, reboiling of Stoddard solvent is employed. The separation between Stoddard solvent and kerosine is readily accomplished with a small amount of steam stripping applied to the kerosine stream.

For crude oils of the type for which this design is most applicable, the recoverable distillates between kerosine and fuel oil residue have no great value other than as cracking stock. For this reason, the secondary flashing operation is designed to recover a bulk distillate as an overhead product with the production of a minimum percentage of fuel oil as residue. Heat for the secondary flashing operation is supplied in the second radiant chamber of the double-chamber heater.

A feature of this plant design is the use of a partial condenser on the bulk distillate from the second flashing operation which is cooled by exchange with partially preheated crude. The temperature of the crude used as a cooling medium in this unit is closely controlled so that there is no possibility of obtaining tube-wall temperatures low enough to allow condensation of steam vapors passing through this condenser. Oil vapors are almost completely condensed and are separated from the uncondensed steam and residual oil vapors. Condensation of the resulting vapor mixture is accomplished in a spray or shower type condenser by direct contact with water. By the use of such a system the effect of corrosion resulting from mild cracking in the second heating operation is concentrated at one point, the direct-contact condenser. Because of the simplicity of design of this condenser, the cost of construction using corrosion-resistant materials is not appreciably greater than that for normal construction. Addition of neutralizing agents at this point can be done easily if desirable. Separation of water and condensate from this direct-contact condenser is carried out in a separating tank.



COURTESY, THE LUMMUS COMPANY.

Unisol Mercaptan Extraction Process

UNIVERSAL OIL PRODUCTS COMPANY

FOR many years petroleum-process development touched only lightly on gasoline-treating methods. Within the last few years demand for higher octane numbers and increasing use of high-sulfur crudes has focused attention on this problem. Removal of sulfur compounds from gasoline gives marked improvement in tetraethyl-lead susceptibility, although the unleaded octane number may improve only slightly. Conventional gasoline-sweetening processes convert malodorous mercaptan sulfur compounds to corresponding alkyl disulfides without reducing sulfur content.

The Unisol process was developed to extract mercaptan compounds from gasoline, thereby accomplishing the purpose of conventional sweetening and at the same time improving tetraethyl-lead susceptibility by removal of the sulfur associated with the mercaptan. Conventional sweetening never improves octane number and often reduces it, particularly with high-mercaptan stocks, making sweetening a nonprofitable operation. Unisol, on the other hand, generally shows a rapid return on the investment through improved product quality and tetraethyl-lead savings. The process is the culmination of years of large-scale pilot-plant development work by The Atlantic Refining Company. It is licensed by Universal Oil Products Company.

The Unisol process is based upon the large increase in solubility of mercaptans in caustic soda solutions when an organic solvent, methyl alcohol, is present.

Hydrogen sulfide-free gasoline is introduced to the bottom of an extraction column and has mercaptan extracted from it as it flows upward in countercurrent contact with an aqueous solution of methanol and caustic soda. The caustic solution is introduced at the top of the extractor and methanol near the mid-point. Above the methanol inlet the caustic solution extracts any methanol dissolved by the gasoline from the caustic - methanol solution. Treated gasoline is removed from the top of the extractor and sent to storage without any subsequent treating except addition of inhibitor. The fat caustic-methanol solution containing extracted mercaptan is removed from the bottom of the extraction column from whence it is charged to a column in which the methanol and mercaptan are stripped from the caustic solution. Regenerated caustic solution is recovered from the bottom of this column for reuse. From the top of the stripping column, stream-methanol-mercaptan vapors go to a condenser and then to a mercaptan separator from which the extracted mercaptans are decanted from the system. Aqueous methanol solution is charged from the mercaptan separator to a fractionating column from which methanol is recovered for reuse as an overhead product.

The process is outstanding in its simplicity, low cost and effectiveness. Mercaptan reduction of better than 99 percent is readily accomplished. Commercial units are operating profitably on gasolines containing as low as 0.013 and as high as 0.25 percent mercaptan sulfur. Octane number improvements at the 3-cc-tetraethyl-lead level, range from 1 to 3 or more on the commercial units, depending on mercaptan-sulfur content of the untreated gasoline.

The treated product as it comes from the unit usually is sweet or nearly sweet. In almost all cases the product is completely sweet after a few days in storage.

The process is noteworthy in its ability to control the gasoline-phenol content. Phenols can be left in the gasoline or can be almost completely removed as a concentrated sodium phenolate solution containing no free sodium hydroxide.

In comparisons of the properties of Unisol-treated gasoline with the raw gasoline or with the product obtained by conventional sweetening, it generally is found that Unisol treating improves inhibitor susceptibility, lowers ASTM gum, and that copper-dish gums after inhibiting are considerably lower.

The ability of the process to extract mercaptans with a circulation of only 3-5 percent caustic-methanol keeps chemical consumption, pumping and utility costs for regeneration of reagents low. Combined chemical and utility costs average about 1 to 1½ cents per barrel treated for most gasolines.

The possibility of a return on the investment usually makes Unisol treating more attractive to install than a conventional sweetening unit. Many gasolines contain more mercaptan than a conventional sweetening unit will successfully handle, making it necessary to remove part of the mercaptan by regenerative caustic extraction. In these cases a Unisol unit will cost no more to install than the combined caustic extraction plus sweetening units.

Many Unisol units ranging in designed capacity from 500 to 25,000 barrels per day now are operating and amply paying their way in improved product quality and reduced tetraethyl lead requirements.

UNISOL MERCAPTAN EXTRACTION UNIT
COURTESY, UNIVERSAL OIL PRODUCTS COMPANY
METHANOL FRACTIONATING COLUMN
CONDENSER
STEAM SUPERHEATER
METHANOL RECEIVER
METHANOL REFLUX PUMP
METHANOL WATER PUMP
STEAM
FUEL GAS
STEAM
MERCAPTAN SEPARATOR
CONDENSER
WATER TO SEWER
REGENERATOR
MERCAPTANS TO DISPOSAL
SETTLER
EXCHANGER
CAUSTIC FEED PUMP
TREATED GASOLINE TO STORAGE
MERCAPTAN EXTRACTOR
CAUSTIC PREWASH
UNTREATED GASOLINE

UOP Catalytic Dehydrogenation Process

UNIVERSAL OIL PRODUCTS COMPANY

THE UOP Catalytic Dehydrogenation Process, broadly speaking, covers the removal of hydrogen from hydrocarbons to produce less saturated hydrocarbons. Such hydrocarbons, depending upon the degree of unsaturation, are useful in the production of engine fuels and as basic materials in the synthesis of rubber, resins and various chemicals. Catalytic butane dehydrogenation means removal of hydrogen from butane to form butylene and hydrogen.

The catalytic butane dehydrogenation process produces large yields of butylene due to the high efficiency of the catalytic reaction. The catalyst speeds up the desired primary reaction of dehydrogenation without a corresponding increase in the reactions which produce undesired secondary products.

The reaction is carried out in reactor tubes containing a suitable catalyst at temperatures ranging from approximately 1000° F. to 1100° F. and pressures ranging from approximately 10 to 50 psi.

The dry normal butane charge is heated to reaction temperature and introduced into the catalyst zone where it undergoes dehydrogenation, drawing the required heat of reaction from an external-circulating flue-gas system.

The material leaving the catalyst tubes is composed of unreacted butane, butylene, hydrogen, and a small percentage of other light gases such as methane, ethane, ethylene, propane, and propylene. It passes through the butane-charge heat exchanger into a gas scrubber where it is cooled by a circulating oil stream and washed free of any entrained tar or coke. The gas then is compressed to 100-150 psi and discharged to the gas-recovery system for separation of the butane-butylene fraction from the hydrogen and light gases.

In order that the processing may be continuous, the reactors containing the catalyst are built in pairs, and by means of suitable, automatically-controlled switch valves, one reactor is kept in service dehydrogenating butane, while the other is being reactivated by having the carbon burned off.

The reactivating system is similar to the processing system and runs parallel with it. Its purpose is to furnish a reactivating gas of controlled oxygen content and sufficient volume to permit burning the carbon from the catalyst without reaching excessively high temperatures.

The reactivating gas leaves the compressor and passes through a heat exchanger and heater; it is then directed by means of switch valves to the catalyst tubes in the spent reactor. The inlet temperature is automatically controlled by firing valves on the reactivating-gas heater and is normally carried at 800° F.

The burning of the carbon from the catalyst produces heat which must be dissipated. Part of this heat is removed by raising the temperature of the reactivating gas itself, the remainder by the external flue gas circulating system.

The reactivating gas leaving the catalyst tubes passes through a heat exchanger and into a gas washer, where it is cooled by water and then returned to the compressor for recirculation. Excess gas, corresponding to the air added, must be discharged from the system in order to prevent a pressure rise on the system. This gas is released by means of a pressure-control valve which maintains constant pressure on the system.

The oxygen content of the reactivating gas is controlled between 1 and 3 percent to avoid excessive temperatures during the carbon-burning period. This is accomplished by means of an oxygen controller, which takes a continuous sample of the circulating reactivating-gas stream and determines the oxygen content. As this varies, the controller operates the inlet-air valve to admit the amount of air necessary to maintain the oxygen content at the desired figure.

The circulating flue gas system, which serves to supply heat to the processing reactors and remove heat from the reactivating reactors, is composed of a blower, a heater, a stack, switch dampers and interconnecting ductwork. Part of the circulating flue gas passes from the blower through the heater, where it is mixed with hot combustion gases to bring it to the desired temperature, after which it passes in indirect heat exchange with the processing reactor. The other part of the circulating flue gas passes through the cooling duct, where it is either sprayed with water or mixed with cool air; it then passes in indirect heat exchange with the reactivating reactor. After passing through the reactors, the heating and cooling flue gases combine in a common outlet duct and return to the blower. Pressure on the circulating flue gas system is controlled by discharging excess gas to the stack.

The commercial catalytic dehydrogenation of normal butane is usually carried out at a conversion of about 30 percent per pass in a recycle operation. The ultimate yield of butylene is 75 to 80 barrels per 100 barrels of butane converted.

In practice, the butane-butylene fraction usually has been fed to an alkylation or a polymerization unit in which the butylenes are converted to alkylate or polymer for the production of high-octane gasoline, while the unreacted butane is recovered for recycling to the dehydrogenation unit.

The direct operating costs for the catalytic dehydrogenation of 1000 barrels daily of normal butane are approximately as follows:

ITEM	Cents per Gallon of Butylene
Operating labor, supervision, laboratory, etc.	.95
Maintenance and materials	.60
Utilities	.70
Taxes, insurance, administration and general overhead	.70
Catalyst	.20
Royalty	.50
	3.65

The above estimated costs cover the dehydrogenation step only. Where further concentration of the butylenes is required, additional operating costs would be involved.

BUTANE CHARGE
GUARD TUBE
BUTANE HEAT EXCHANGER
BUTANE HEATER
REACTOR "B" (REACTIVATING)
REACTOR "A" (PROCESSING)
SWITCH VALVES
REACTIVATING GAS HEATER
R.G. EXCHANGER
TO DRAIN
R.G. WASHER
TO VENT
R.G. COMPRESSOR
AIR COMPRESSOR
WASHER WATER
AIR
PROCESS GAS SCRUBBER
SCRUBBER OIL COOLER
SCRUBBER OIL PUMP
PROCESS GAS COMPRESSOR
ABSORBER BOTTOMS
GAS TO ABSORBER
RECEIVER
PRECOOLER
LIQUID TO STRIPPER
U.O.P. CATALYTIC DEHYDROGENATION PROCESS
COURTESY, UNIVERSAL OIL PRODUCTS COMPANY

UOP Catalytic Polymerization Process

UNIVERSAL OIL PRODUCTS COMPANY

THE UOP Catalytic Polymerization Process is one of the first of the many catalytic processes presently being employed in the petroleum-refining industry. The development of the catalytic polymerization process offers to the petroleum refiner a means for converting a portion of fuel gas produced in cracking operations into a high-grade motor gasoline, which increases the yield of gasoline obtainable from each barrel of crude oil processed. The process, in effect, then produces a finished gasoline of high value from a raw material which has the lowest value.

Most generally the process is used to polymerize propylene and butylenes found in the gases produced from thermal or catalytic cracking of topped crudes or gasoils. Propylene and butylenes can be polymerized either alone or in admixture, depending on the type of product desired.

Polymerization of the butylenes in the absence of other olefins can be controlled to yield a product which on hydrogenation will rate 90-95 octane number. This is referred to as a selective polymerization operation used to produce aviation-gasoline components. Polymerization of propylene and butylenes in a mixture for the production of a fuel which will be blended in motor gasoline is referred to as a nonselective operation. This is the most common of all polymerization operations.

Propylene can also be polymerized separately to make either a motor polymer or a product rich in either C_6, C_9 or C_{12} polymer, depending on operating conditions. In recent years the butylenes normally found in refinery gases have in some cases become more valuable when converted to other products and consequently many plants are being operated for the polymerization of the residual propylene.

Briefly, the polymerization operation is one which consists of contacting the olefin-containing feed stream with the catalyst at temperatures in the range of 350° F. to 475° F. and at pressures ranging from 150 to 1200 psi. Processing conditions vary with the type of plant employed, with the feed stock processed and with the type of product desired.

There are two types of units presently being used for carrying on the process of polymerization. The most commonly used design is referred to as a chamber type. In this unit the catalyst is contained in a vertical chamber. Catalyst is loaded in one or more chambers, each containing multiple catalyst beds of 5 feet to 10 feet in depth.

Since the polymerization reaction is exothermic and the system in which the reaction takes place is essentially adiabatic, an increase in the temperature of the reactants results as they travel through the catalyst beds. In order to maintain the catalyst temperatures in the range considered most effective for good olefin conversion and catalyst life, it is desirable to control the amount of reaction that occurs in the catalyst chamber by the recycling of hydrocarbons which have been through the catalyst chambers. This recycle material, being low in olefin content, will act to dilute the olefins in the fresh feed. It is possible in this manner effectively to limit the temperature rise through the reaction zone.

The other, or reactor type of unit, employs heat-exchanger type reactors. In this design the catalyst is loaded in vertical small-diameter tubes which are surrounded with a cooling medium to remove the heat of reaction.

Reaction temperature control is therefore obtained by controlling the temperature of the cooling medium, and in this manner the reaction approaches isothermal conditions. In this unit it is therefore possible to process feeds of high olefin content without necessity for the use of recycle as in the case of the chamber-type unit.

Reactor-type units are most commonly used when the feed to be processed is high in olefin content such as is the case for C_3 and C_4 fractions from catalytic cracking. Although the reactor installation is somewhat more complicated than the simple catalyst chamber, a compensating saving in plant size is effected by eliminating the need for handling a recycle stream which can appreciably increase the total plant throughput in those cases where feed olefin contents are high.

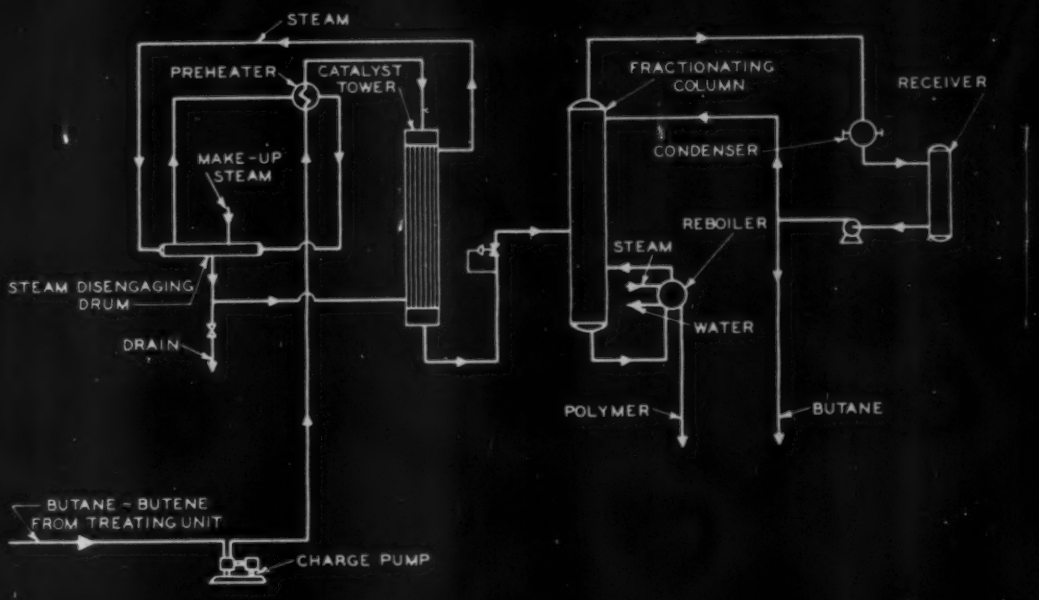
Catalytic Polymer Properties

Type of Plants	Chamber	Reactor	Reactor
Pressure psig	500	1000	1000
Temperature °F.	370-365	400	450
Feed Stock	Cracking Plant Stabilizer Net Overhead Liquid	Butane-Butylene from Refinery Gas Concentration System	Propane-propylene from Refinery Gas Concentration System
Polymer Inspection:			
Gravity	64.0	64.3	62.4
IBP	96	78	144
10 Percent	156	152	204
30 Percent	210	238	244
50 Percent	235	258	266
70 Percent	279	....	284
90 Percent	366	379	330
EP	430	416	402
Rec.	97	95	....
RVP	9	11	4
Average Polymer Properties:			
Bromine No.	130-150		
Induction Period:			
Clear	15 min.		
+ 0.025 Percent UOP No. 1	500-550		
+ 0.005 Percent UOP No. 4	450-500		
Copper Dish Gum	25 mgs.		
ASTM Octane Number Clear		82.5	
ASTM Octane Number +3 cc TEL		85.0	
Research Octane Number Clear		97.0	
Research Octane Number +3 cc TEL		100.0	
Polymer Blending Values:			
5-10 Percent Blends in Average Straight Run Gasoline			110-120
5-10 Percent Blends in Thermal Cracked Gasoline			95-100

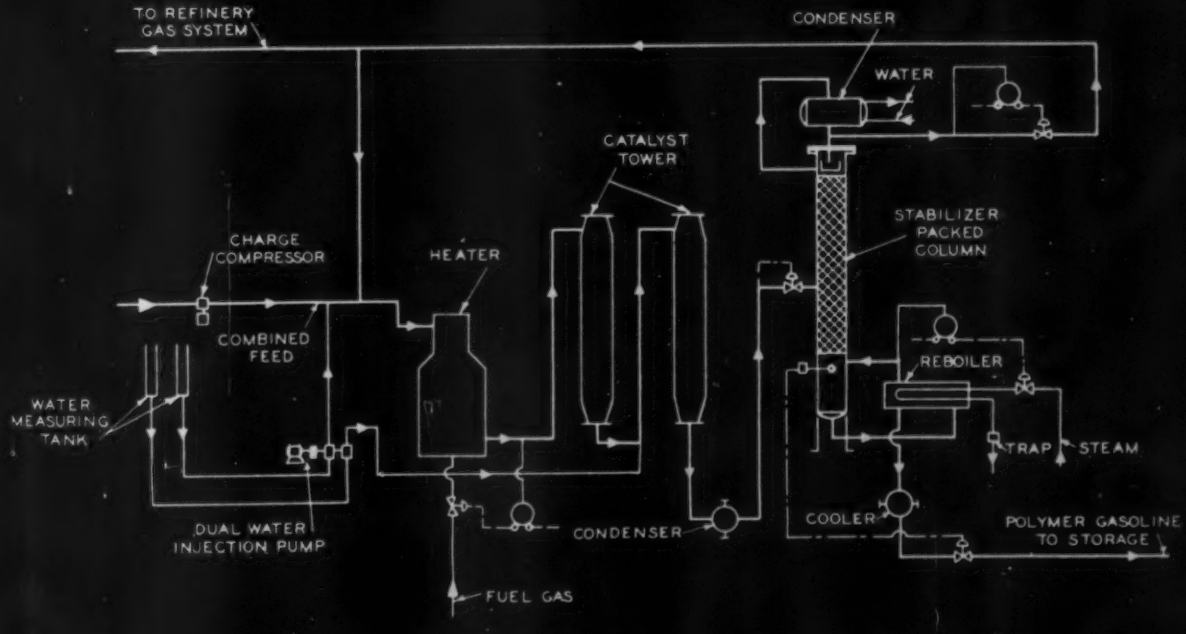
Following is an approximate utility requirement for a chamber-type polymerization unit producing 100 barrels per day of polymer:

Steam*	2500 Lbs./Hr.
Water	250 GPM
Electricity	15 KW

* Steam requirement can be reduced in proportion to amount of hot oil available for heat exchange.



U.O.P. CATALYTIC POLYMERIZATION UNIT - REACTOR TYPE



U.O.P. CATALYTIC POLYMERIZATION UNIT - CHAMBER TYPE

COURTESY, UNIVERSAL OIL PRODUCTS COMPANY

UOP Isomerization Process

UNIVERSAL OIL PRODUCTS COMPANY

THE UOP Isomerization Process was developed for the isomerization of normal paraffins to isoparaffins. This process has been used extensively for the isomerization of normal butane to isobutane which is one of the raw materials for making high-octane-gasoline blending component known as alkylate. The process is also applicable for the isomerization of normal pentane and hexane fractions for improvement of the octane number of these fractions.

The process involves the contacting of a dry feed with a catalyst which contains aluminum chloride and hydrogen chloride. The catalyst is deposited on the quartz-chips packing in the reaction vessel and the activity of the catalyst is maintained at the desired level by the continuous introduction of fresh aluminum chloride dissolved in a portion of the liquid charge. Anhydrous hydrogen chloride is used as a promoter for the reaction and this material is recycled within the system to maintain the required concentration in the reaction zone.

The liquid hydrocarbon and chemicals composing the combined feed are contacted in their passage through the reaction zone. In the isomerization reaction a minor secondary reaction takes place, namely, the formation of a viscous liquid sludge containing dissolved aluminum chloride and hydrogen chloride which spreads over the packing surfaces. The sludge thus formed continuously drains from the packing and separates from the butane stream below the catalyst zone in the bottom section of the reactor, and this small amount of material is drawn off intermittently to a neutralizing pit. The hydrocarbon effluent from the reaction zone, containing dissolved aluminum chloride and hydrogen chloride, then enters the aluminum chloride and hydrogen chloride recovery section.

The isomerization reaction is slightly exothermic. The heat generated in the process is absorbed by the vaporization of some of the liquid passing through the reactor, and as a result the reaction is essentially isothermal. The process is operated at pressures in the order of 250-350 psi and temperatures of 190° F. to 220° F.

The effluent from the reactor is fed to an aluminum chloride separation column where the net hydrocarbon product, together with recycled hydrogen chloride is separated from the aluminum chloride which is concentrated in the bottoms stream from the column, consisting mainly of pentanes and normal butane.

The hot stream from the bottom of the aluminum chloride column containing between 1 and 3 pounds of aluminum chloride per barrel is returned to the top of the reaction vessel. Recycling aluminum chloride in this manner makes it possible to maintain a high concentration of aluminum chloride in the reactor which could not otherwise be possible without excessive chemical consumption. A small portion of the bottoms stream from the aluminum chloride column is withdrawn to a caustic-wash tower to reject continuously the small amount of pentanes formed in the process, which otherwise would build up in the system.

The overhead from the aluminum chloride column is fed to a conventional HCl stripper to separate the HCl from the net plant product. The HCl from the top of the HCl stripper is returned to the reactor as a recycle stream. The bottoms stream from the HCl stripper is caustic washed to remove any traces of hydrogen chloride before leaving the unit.

The net product from the plant consisting of a mixture of isobutane and normal butane is sent to a deisobutanizer where the unconverted normal butane is separated and returned to the isomerization unit. Since the isomerization unit normally operates in conjunction with an alkylation unit, the isobutane-normal butane separation generally is carried out in the fractionation train of the alkylation unit which separates an isobutane-rich stream which in turn is fed to the alkylation reactors.

The isomerization of pentanes or hexanes is carried out in substantially the same manner as that described for the processing of normal butane with the exception that in the processing of these heavier materials there is a greater tendency for the formation of sludge, and this must be counteracted by the injection of an inhibitor which serves to suppress secondary sludge-forming reactions.

Typical Isomerization Results
Normal Butane, Pentane and Hexane Feeds

TYPE OF FEED	n-Butane	n-Pentane	Hexane Fraction
Operating Conditions:			
Reactor Temperature, °F.	190	180	180
Reactor Pressure, psig.	250	250	250
HCl in Reactor Feed Mol., Percent	7-10	7-10	7-10
Product Analyses:			
Isobutane	50	....	....
Normal Butane	48	....	....
Isopentane		60	
n-Pentane	2	38	
Hexane and Heavier		2	
Octane Ratings:			
Feed ASTM O.N. Clear			58-60
Product ASTM O.N. Clear			75-80
3 cc TEL/gal.			88-93
Catalyst Consumption:			
Lbs. $AlCl_3$/bbl. iC_4 or iC_5 produced	0.3-0.5	0.5-0.7	
Lbs. HCl/bbl. iC_4 or iC_5 produced	0.05	0.05	

Based on processing to produce 450 barrels per stream day of isomerized product, normal utility requirements would be approximately as follows:

Steam	8000-9000 lbs./hr.
Cooling water	600- 800 gpm
Electricity	20 kw

NORMAL BUTANE, PENTANE OR HEXANE FRESH FEED
RECYCLE
REACTOR
HCL RECYCLE
ALUMINUM CHLORIDE SATURATOR
ALUMINUM CHLORIDE TOWER
SLUDGE
HCL COLUMN
ISOMERIZED PRODUCT
CAUSTIC
SPENT CAUSTIC
U.O.P. ISOMERIZATION PROCESS
COURTESY, UNIVERSAL OIL PRODUCTS COMPANY

UOP Paraffin Alkylation Processes

UNIVERSAL OIL PRODUCTS COMPANY

ALKYLATION of paraffins is a reaction in which an isoparaffin is combined with an olefin to produce a branched-chain paraffin hydrocarbon of higher molecular weight known as alkylate.

Alkylate is a mixture of saturated, stable liquid isoparaffinic hydrocarbon that boils in the gasoline range. It has an ASTM octane number in the range of 88-93 depending on the conditions under which it is made and the character of the feed.

Extensive use was made of the alkylation process during the war to produce a high-octane-number component of aviation gasoline. Although initially considered as a high-cost operation, many simplifications have been achieved in the process which make it attractive for producing a high-octane gasoline-blending component for improved-quality motor fuels.

The operation of this process is particularly attractive in those cases where low-cost isobutane is available in sufficient quantities to alkylate with available olefins. Although some isobutane is present normally in admixture with olefins in gases produced from thermal-cracking operations, it is not available in sufficient quantities to alkylate with the available olefins. In these cases isobutane must be made up from other sources such as separation from natural gas or isomerization of available normal butane.

Light gases from catalytic cracking will in some cases, depending upon the conversion and type of catalyst employed, contain a relatively high percentage of isobutane, sufficient in quantity to alkylate with a substantial portion of the butylenes and propylenes available. In such instances the alkylation process is well suited to operate in conjunction with catalytic cracking to obtain the maximum yield of high-octane gasoline.

The UOP Paraffin Alkylation Process is a catalytic process utilizing either hydrofluoric acid or surfuric acid as the catalyst. The materials which enter into the alkylation reaction are isobutane and olefins. The olefins ordinarily employed are butylene, propylene, and amylene. These olefins can be reacted with isobutane individually or collectively, depending on their availability.

When isobutane is alkylated with butylenes, the volume of isobutane should be approximately 20 percent greater than the volume of olefin, yielding 160 percent by volume of rerun alkylate.The yield of alkylate and the consumption of isobutane will vary slightly, depending on the type of olefin alkylated.

In order to favor the alkylation reaction and minimize side reactions such as polymerization, the feed to the alkylation reactor is provided with an excess of isobutane over and above that required for alkylating the olefins. This is accomplished by recycling a stream rich in isobutane. The higher the ratio of isobutane to olefin in the feed to the reaction zone, the higher the yield and quality of the alkylate produced. Economic considerations and product quality requirements will determine the ratio used. For most cases the recycle of isobutane should be such as to provide five to ten times as much isobutane as olefin in the total feed to the reactor.

The alkylation reaction is carried out at temperatures in the range of 45° F. to 100° F. The lower temperatures are employed when using sulfuric acid, since operating temperature influences product yield and quality as well as catalyst life. This relation does not apply when hydrofluoric acid is used; therefore, the reaction may be carried at the higher temperature when using this catalyst. The operating pressure is such as to maintain the reaction mixture in a liquid phase.

The reactor system used for this process provides for the intimate mixing of the dry hydrocarbon feed with the catalyst as well as cooling of the reaction mixture to remove the heat liberated by the reaction.

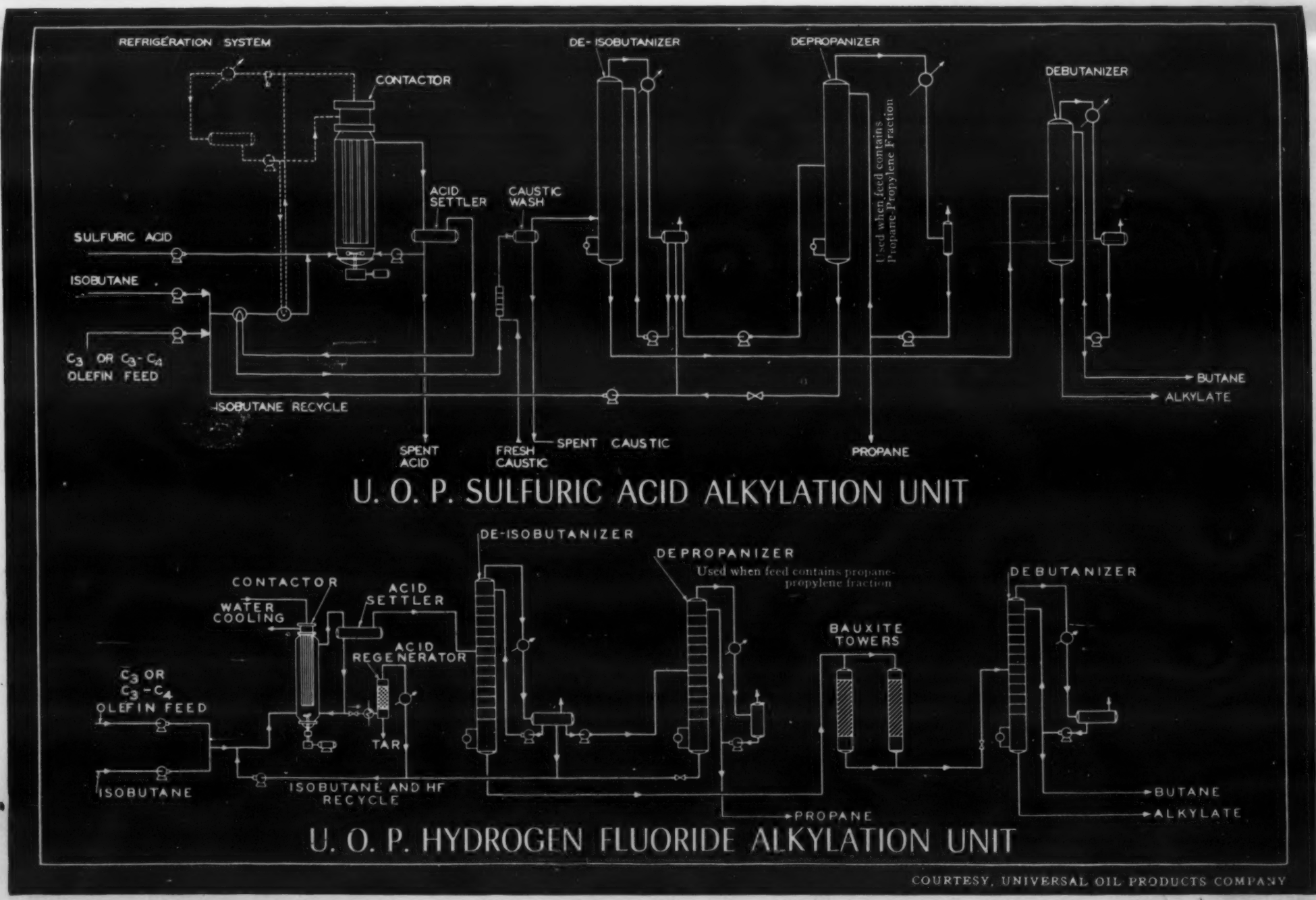
After completion of the reaction in the reaction zone, it is necessary to separate the total hydrocarbon reaction mixture from the catalyst as well as to separate the alkylate product from isobutane recycle and the hydrocarbons, such as normal butane and propane, which do not enter into the reaction.

Because of the difference in physical characteristics of the hydrofluoric acid and sulfuric acid catalysts which may be used for promoting the reaction, the design of the unit will differ somewhat in the methods for contacting and separating the catalyst from the hydrocarbon mixture, depending on the catalyst used.

In the alkylation process one of the important operating variables is the strength of the acid. Since the acid catalyst in service becomes contaminated with dissolved tars, it is necessary to continuously replace a portion of the acid catalyst with fresh material to maintain a desired level of acidity. When sulfuric acid is used as the catalyst the acid strength is maintained by introducing fresh high-strength acid and discarding spent acid, which can be used for other operations in the refinery, such as gasoline treating. Since hydrogen fluoride has a vapor pressure slightly higher than isopentane, it can be purified by a distillation step to effect the separation from the heavy hydrocarbons. The hydrofluoric acid purification is carried out on a continuous basis, returning the purified acid to the reaction zone to maintain the desired acid strength.

Catalytic Alkylation—Typical Results

FEED STOCKS	Butylenes from Catalytic Cracking	Amylenes from Catalytic Cracking	Propylene
Operating Conditions:			
Isobutane-Olefin Ratio	5:1	10:1	10:1
Reaction Temp.—°F. HF	90	60	45
Reaction Temp.—°F. H_2SO_4	45	45	90
Acid-Hydrocarbon Ratio	1:1	1:1	1:1
Acid Strength	87-90	87-90	87-90
Product Quality:			
Octane Number ASTM Clear	92	89	85
E.P.	350	350	350
RVP	4.5	2.3	2.0
Conversion Factors:			
Vol. 350° EP Alky./Vol. Olefins	1.6	1.6	1.6
Vol. 350° EP Alky./Vol. Isobutane	1.25	1 1	1.1
Catalyst Consumption:			
Using HF as catalyst—Gallons Alkylate/lb Acid	85	60	60
Using H_2SO_4 as Catalyst—Gallons Alkylate/lb Acid	2.0	0.5	0.5


REFRIGERATION SYSTEM
CONTACTOR
ACID SETTLER
CAUSTIC WASH
DE-ISOBUTANIZER
DEPROPANIZER
DEBUTANIZER
Used when feed contains Propane-Propylene Fraction
SULFURIC ACID
ISOBUTANE
C3 OR C3-C4 OLEFIN FEED
ISOBUTANE RECYCLE
BUTANE
ALKYLATE
SPENT ACID
FRESH CAUSTIC
SPENT CAUSTIC
PROPANE
U. O. P. SULFURIC ACID ALKYLATION UNIT
DE-ISOBUTANIZER
DEPROPANIZER
Used when feed contains propane-propylene fraction
DEBUTANIZER
CONTACTOR
WATER COOLING
ACID SETTLER
ACID REGENERATOR
BAUXITE TOWERS
C3 OR C3-C4 OLEFIN FEED
TAR
ISOBUTANE
ISOBUTANE AND HF RECYCLE
BUTANE
ALKYLATE
PROPANE
U. O. P. HYDROGEN FLUORIDE ALKYLATION UNIT
COURTESY, UNIVERSAL OIL PRODUCTS COMPANY

UOP Thermal Cracking Process

UNIVERSAL OIL PRODUCTS COMPANY

THE thermal cracking process is probably the best known process employed by the oil-refining industry. It is of the widest application, both geographically and practically. Thermal cracking is in successful use in every country where oil is refined, and it is producing good gasoline from every variety of charging stock, from naphthas to the heaviest crudes and residual oils.

Universal Oil Products Company invented and developed the first cracking process that could convert heavy oils to produce maximum yields of gasoline. For more than 25 years this company's engineers have been designing, servicing and improving cracking equipment in order to make the process more serviceable to refiners of both large and small capacity.

Thermal cracking produces butylenes and propylene which can be polymerized to produce polymer gasoline of high octane number for use as a blending fluid to improve the quality of other gasolines.

The ideal equipment for thermally cracking topped or reduced crudes is centered about the two-coil cracking unit designed for optimum results when producing high-grade cracked fuel oil residuum.

The two-coil cracking unit is shown in the accompanying flow diagram.

The topped or reduced crude charge is introduced to the side of the fractionating column where it is contacted with the vapors from the flash chamber. The overhead product from the fractionating column is end-point gasoline. Furnace or tractor distillates are withdrawn as side cuts and stripped to specifications. From a lower side-cut tray a higher-boiling distillate comprising the lower-boiling fractions of both the recycle and charging stocks is withdrawn as charge to the light-oil heater. The charge to the heavy-oil heater is withdrawn from the bottom of the fractionating column, and consists of the highest-boiling fractions of both the recycle and charging stocks.

The transfer lines from the two heaters enter the top of a downflow reaction chamber. This chamber is operated without a liquid level, all products being withdrawn from the bottom through a pressure control valve.

The mixture leaving the reaction chamber after suitable quenching enters the flash chamber in which a fuel oil residuum of the desired properties is separated.

With the development of the U.O.P. Equiflux heater and its incorporation into the two-coil selective plant described above, it is possible to obtain temperatures as high as 920° F. in the middle of the reaction chamber. With a reaction chamber operation at this temperature level the heavy charge is subjected to a far greater time-temperature action than can possibly be achieved without the use of the reaction chamber as a means of viscosity breaking. As a result, yields are improved, and at the same time plant costs are reduced due to the greatly increased cracking rates obtained at high temperatures.

Cracking residual stocks at high temperature levels has a further advantage resulting in cracked fuel oils of maximum stability and minimum sediment content. These properties are direct functions of the average temperature level of cracking, and in the modern two-coil unit the average temperature of the entire reactor chamber is greater than the maximum temperature ordinarily attained in coil-only viscosity breaking.

Similar results to a lesser degree may be obtained in a single coil and reaction chamber unit in which the charge passes through the heater with all of the recycle stock.

To illustrate the comparative results obtained in various methods of processing, the following yields are presented as representative results when charging a typical 25° API Mid-Continent reduced crude having a viscosity of 20 S.F.S. at 122° F. In considering these results, it must be remembered that not only the gravity but also the viscosity of the charge are important in determining yields.

TABLE 1

Yields from 25° API Mid-Continent Reduced Crude in Residuum Operation

	Maximum Gasoline	Maximum Furnace Oil
Percent by volume of charge:		
Gasoline, 400 E.P., 10 RVP	53.5	38
Furance Distillate	....	23
Fuel Oil	37.5	34
Liquid volume loss	9	5
Gas, cu. ft/bbl.	500	290
Octane number (A.S.T.M.)	69-70	68-69
Fuel Oil, vis. S.F.S. at 122 °F.	200	200
Fuel Oil, gravity, °API	7	8
Percent Catalytic Polymer Gasoline, 10 RVP	5	3.5
Percent Total Gasoline, including Polymer	58.5	41.5
Octane Number of Total Gasoline	71-72	70-71

It should be noted that the cracked gasoline yields of Table 1 are reported on the basis of 400° F. end point and 10-pound Reid vapor pressure. On the basis of total butane-butylene retention in the gasoline the yields would be increased approximately 4 percent and 3 percent, respectively. The yields of catalytic polymer gasoline shown in the table are on the basis of polymerizing 80 percent of the propylene and butylenes formed in the cracking operation.

ABSORBER
STABILIZER
STABILIZED DISTILLATE
FRACTIONATOR
CHARGE
CHARGE FOR VIS-BREAKING OPERATION
RESIDUUM
RESIDUUM FLASH COLUMN
FLASH CHAMBER
REACTION CHAMBER
HEATERS
FUTURE REFORMING CHARGE
U.O.P. THERMAL TWO-COIL SELECTIVE CRACKING PROCESS
COURTESY, UNIVERSAL OIL PRODUCTS COMPANY

UOP Thermal Reforming Process

UNIVERSAL OIL PRODUCTS COMPANY

THE UOP Thermal Reforming Process has had such wide and successful application in all parts of the world that it ranks with thermal cracking as one the best known processes in the petroleum-refining industry. This process converts low-grade gasolines and naphthas into high-octane gasoline by subjecting the charging stock to heat treatment under controlled time, temperature and pressure conditions. The process is flexible, not only in its ability to process various stocks, but also as to quality and quantity of products which can be made from those stocks.

Thermal reforming produces butane-butylenes and propane-propylene which can be polymerized to produce a high-octane polymer gasoline which is an excellent blending material for improving the quality of other gasolines. The residuum produced by this operation has a high calorific value, and can be marketed as fuel oil.

As shown by the accompanying flow chart, a straight-run gasoline or naphtha fraction is charged to a heater wherein the reaction time, pressure, and discharge temperature are controlled to produce the desired products. The effluent from the heater passes through a pressure-control valve where the pressure is reduced. A quench stream from the fractionator is introduced after the control valve to reduce the temperature of the effluent and minimize coking.

The quenched products enter a combination flash chamber and fractionating column wherein the gas and gasoline are taken overhead, and the small amount of bottoms produced is removed from the lower portion of the vessel. A cracked-naphtha side-cut from this column is used for quench and heat-exchange duty. Naphtha side-cuts may be withdrawn if desired.

The overhead products from the fractionator are cooled, and the condensed, unstabilized gasoline is sent to a stabilizer to produce the desired vapor pressure. If polymerization of the gases evolved from the cracking operation is desired, an absorber for the receiver gases plus deep stabilization of the gasoline is employed to provide maximum recovery and utilization of polymerizable olefins available.

The bottoms withdrawn from the flash-chamber portion of the reformer column are further flashed in a residuum stripper. Light material removed from the residuum at this point is condensed and returned to the fractionating column, whereas the residuum is sent to storage for subsequent disposal as fuel oil. The properties of the residuum are regulated by suitable control of the reflux to the stripper.

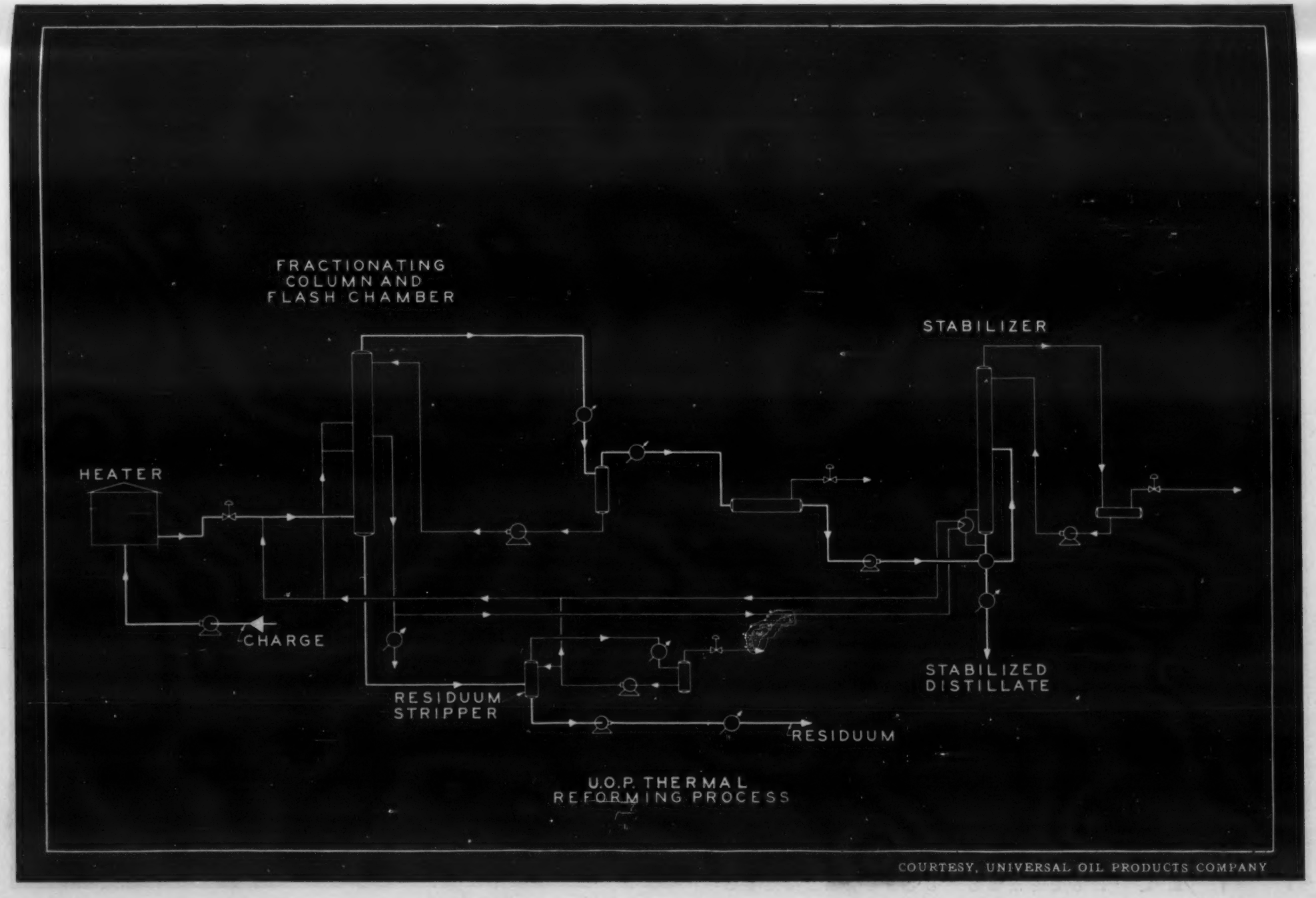
A typical result when reforming a 53° API Mid-Continent straight-run naphtha is shown in the following tabulation. It must be realized in considering these results that the yields and octane-rating improvement that can be derived from thermal reforming are dependent upon the characteristics of the charging stock. In order to design thermal reforming equipment for any refiner, his entire situation must be carefully analyzed and studied, not only from the standpoint of charging stocks available, but also from that of market requirements.

Thermal Reforming of Mid-Continent Naphtha

Charging Stock	
Gravity °API	53.0
Octane rating, ASTM (F-2) clear	40.5
Characterization factor	11.96
100 ml. distillation	
I.B.P. °F.	208
10%	263
50%	314
90%	367
E.P. °F.	389
Products	
Gasoline:	
Yield, vol. % of charge	81.3
Gravity, °API	56.1
Reid vapor pressure, lbs.	10.0
100 ml. distillation	
I.B.P. °F.	93
10%	145
50%	270
90%	360
E.P. °F.	398
Octane Ratings:	
ASTM (F-2), clear	70.2
+ 3 cc. TEL/Gal.	81.9
Research (F-1), clear	78.2
+ 3 cc. TEL/Gal.	91.3
Residuum	
Yield, Vol % of charge	4.2
Gravity, °API	18.2
Gas	
Yield, Wt. % of charge	15.3
Cu. Ft./Bbl. of charge	467

The normal utilities required to operate a thermal reforming unit based upon 1000 barrels per stream day are approximately as follows:

Steam (150 psi.)	1400-200 lb./hr.
Fuel	9-10 × 10^6 Btu/hr.
Cooling water	400-500 GPM
Electricity	10-15 KW



COURTESY, UNIVERSAL OIL PRODUCTS COMPANY

Vapor Recovery Process

FOSTER WHEELER CORPORATION

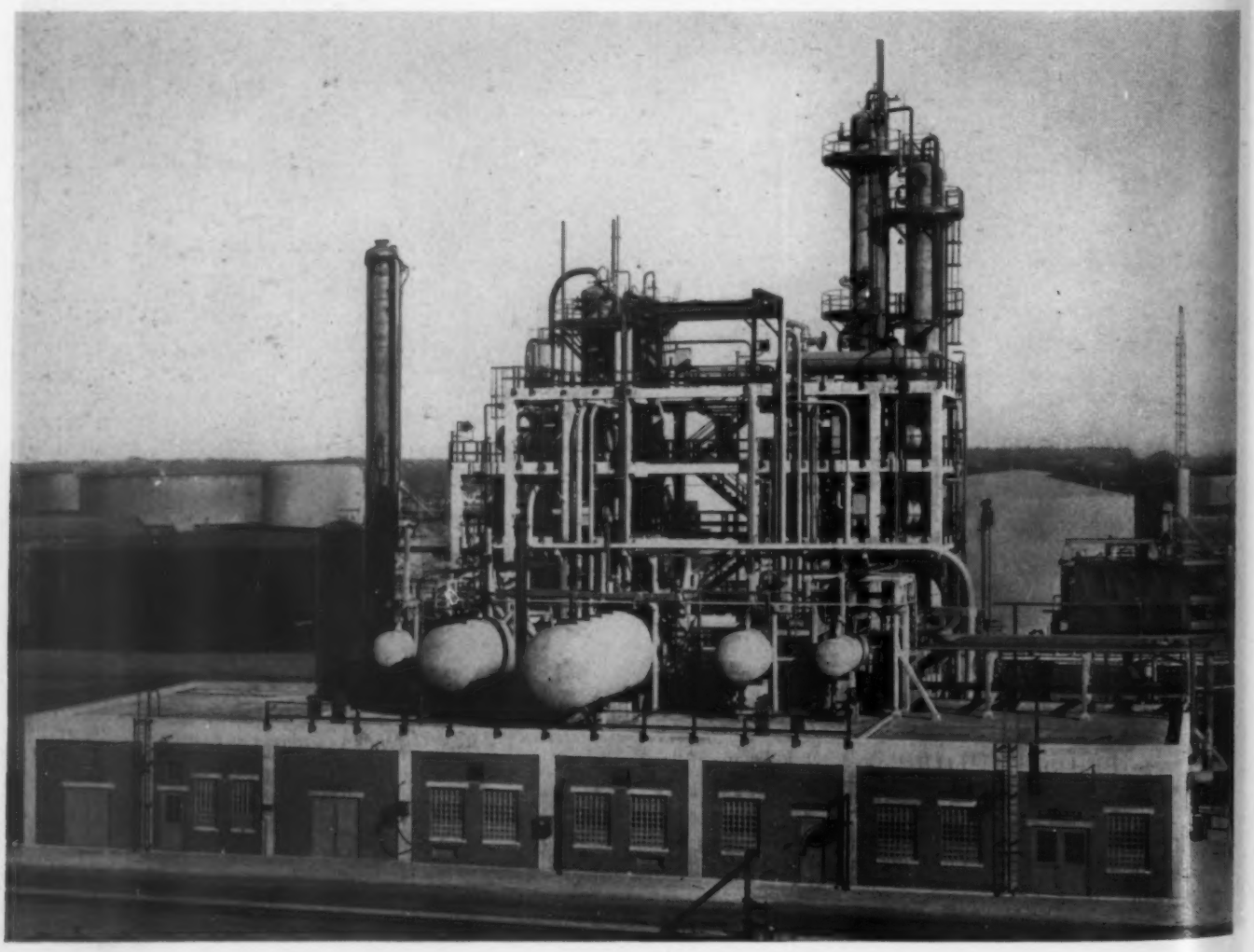
THE three refinery operations, absorption, debutanization, stabilization and reruning were, until recent years, three separate process units and were generally located in different parts of the refinery. However, the modern trend is to combine these processes in one self-contained unit, thereby affecting substantial reduction in labor and utility costs and initial capital expenditure.

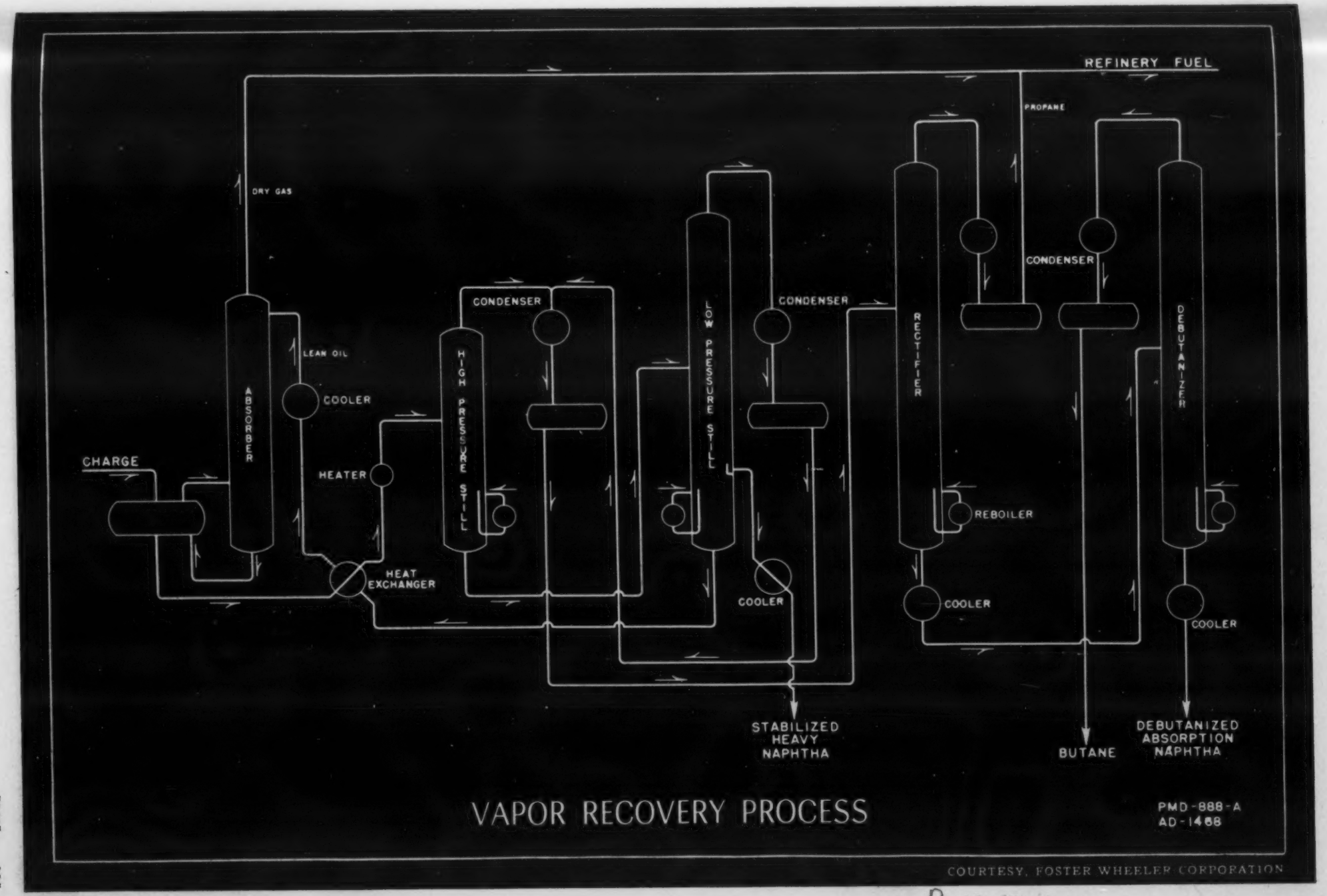
In the diagrammed typical vapor-recovery unit, employed very successfully to process fluid catalytic cracking unit products, the gas and liquid charge is united in the charge tank. C_2's and lighter are removed from the system as gases from the top of the absorber with the heavier gaseous products being absorbed by the lean oil and returned to the charge tank. The total liquid is then charged to a high-pressure still where the naphtha is partially stabilized and charged to the low-pressure still for complete stabilization. The net overhead product from both the high- and low-pressure stills are joined in the high-pressure still accumulator and constitute the charge for the rectifier and debutanizer.

The high- and low-pressure still system is installed in place of a single still in order to avoid the use of high-pressure (600 psig) steam for reboiling.

Under this system the total overhead steam is condensed at 100 psig and the stabilized bottoms product reboiled at 30 psig. This permits the use of 100-psig steam in the reboilers and normal plant cooling water in the condensers. The bottoms product from the low pressure still is a 2-pound R.V.P. gasoline, part of which is used as lean oil in the absorber and recycled to that tower after being cooled to 90° F.

The net overhead product from the still system is charged to the rectifier from which propane is taken as the overhead product. The bottoms from this tower is charged to the debutanizer, the last tower in the vapor recovery system. The overhead product, a butane: butane cut, is a valuable stock for polymerization and alkylation. The bottoms product is a debutanized light gasoline fraction.





COURTESY, FOSTER WHEELER CORPORATION

Depropanizer

Glycol-Amine Gas-Treating Process

THE FLUOR CORPORATION, LTD.

THE glycol-amine process for the purification and dehydration of a gas represents a continuous operation of two established processes. Aqueous solutions of the ethanolamines are capable of absorbing CO_2 and H_2S at atmospheric temperatures. When the solutions containing the acidic constituents are heated to their boiling points, these components are expelled from the solution. Similar use of diethylene glycol in the removal of water vapor from a gas stream led to a study for the combination of these two processes for the purification and dehydration in one unit.

The combination of these two principles as developed by The Fluor Corp., Ltd., provides a process for simultaneous acid gas removal and dehydration wherein the ethanolamine and diethylene glycol are included in the same solution. However, the combination feature, whereby both purification and dehydration may be accomplished in one stage, is not the only advantage of the process. A glycol-amine solution may be regenerated more completely than an aqueous amine solution, thus permitting the attainment of higher purities in the gas being treated. At the same time, a glycol-amine solution with the same water content as a diethylene glycol solution will dehydrate the gas to a slightly greater degree than the latter.

The flow diagram on the opposite page represents a typical application in the treatment of a sour-gas stream. Sour gas is introduced into the bottom of the absorbing column, the contactor. As it passed upward through the bubble trays it is thoroughly contacted with the glycol-amine solution which enters at the top of the vessel. The outlet gas, free of the CO_2 and H_2S and dehydrated, leaves the top of this column. The rich solution containing the absorbed acid gases leaves the bottom of the contactor and passes through a bank of conventional heat exchangers, where the temperature of the solution is raised. The rich solution then enters the regenerating column, the still. As it flows down over the bubble trays in this column it is contacted with the rising stripping vapors, thus stripping the solution of the acidic constituents. The solution from the bottom tray of the still enters a reboiler wherein it is boiled to provide the stripping vapor for the column. The overhead stream of acid gas and water vapor from the still passes through a condenser, and the resultant mixture is separated in an accumulator, the acid gases passing overhead from this vessel and the condensate being returned to the still as reflux. The regenerated lean glycol-amine solution leaves the bottom of the still, passes through the exchangers, and is pumped back through coolers to the top of the contactor, thus completing the cycle.

When using an aqueous monoethanolamine solution it is possible to operate the still at higher than atmospheric pressures and gain somewhat more complete regeneration insofar as CO_2 is concerned, although increased pressure usually is detrimental in a stripping operation. In this particular case the dissociation of the monoethanolamine carbonate increases sufficiently with the increased temperature caused by the additional pressure to overcome the adverse effects of the increased pressure. Such is not the case with H_2S and the stripping of this constituent is less complete at an elevated pressure.

The addition of glycol to the solution increases the boiling point without necessitating an increase in pressure in the regenerating column. Thus, in the case of a glycol-amine solution the adverse features of increased pressure are not encountered, and both CO_2 and H_2S may be stripped completely from the solution.

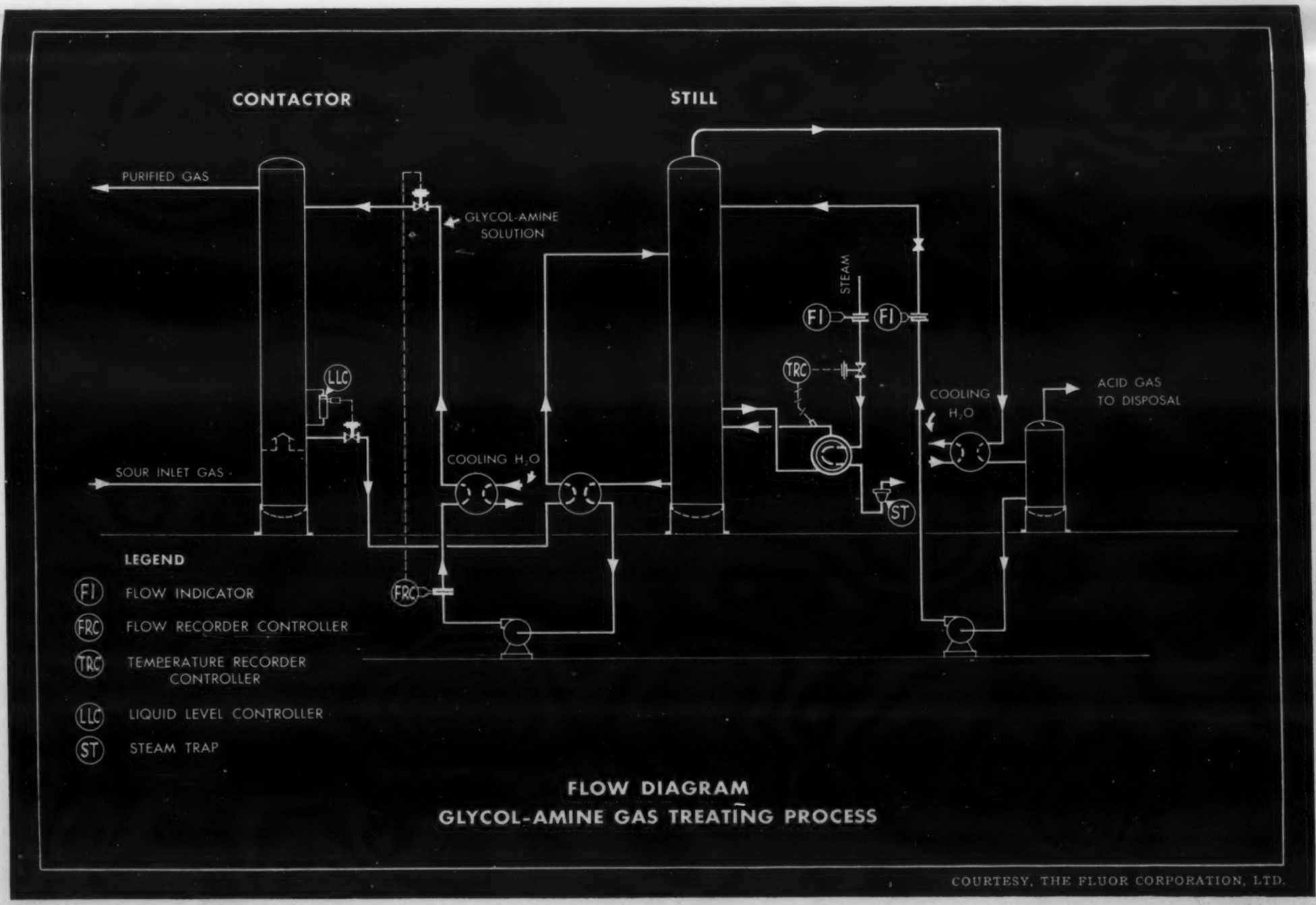
The maximum purity obtainable on any gas is a function of the pressure and temperature at the top of the acid-gas-absorbing column and the amount of acid gas remaining in the stripped solution. It is obvious that higher purities are attainable by the use of the glycol-amine solution than by the use of an aqueous monoethanolamine solution, since the former may be more completely stripped. This is particularly important when considering low H_2S specifications for treated gas. A difference of but a few grains of H_2S in the regenerated solution may mean the difference between meeting and not meeting the specified purity. The fact that glycol-amine solutions may be so easily stripped is one of the major advantages of the process.

Although the process originally was developed to accomplish simultaneous acid-gas removal and dehydration, its application is not limited to this use. If dehydration is not required, the glycol and water contents of the solution may be adjusted so as to meet residue-gas specifications. Adjusting the concentration of these two components allows setting the regeneration temperature to obtain the required degree of acid-gas stripping.

Typical solution compositions employed in the glycol-amine process may be comprised of from 10 to 30 percent monoethanolamine, 45 to 85 percent diethylene glycol, and 5 to 25 percent water. The ability of adjusting components of the glycol-amine-water solution to attain required results permits flexible operation. A wide range of specifications for treated gas purity and water content may be met by using this process. Sour natural gas may be treated readily so that the residue gas will contain less than 0.25 grains of H_2S per 100 cubic feet, which is less than 0.0004 mol-percent of H_2S in the gas.

Although the glycol-amine process was originated for simultaneous purification and dehydration of gas, its use for acid-gas removal only is constantly increasing with the more rigid specifications in gas purity. Consequently, the full contribution of this process to industry is yet to be realized.

This description of the process and the flow diagram opposite were provided by The Fluor Corporation, Ltd., Los Angeles.



FLOW DIAGRAM
GLYCOL-AMINE GAS TREATING PROCESS

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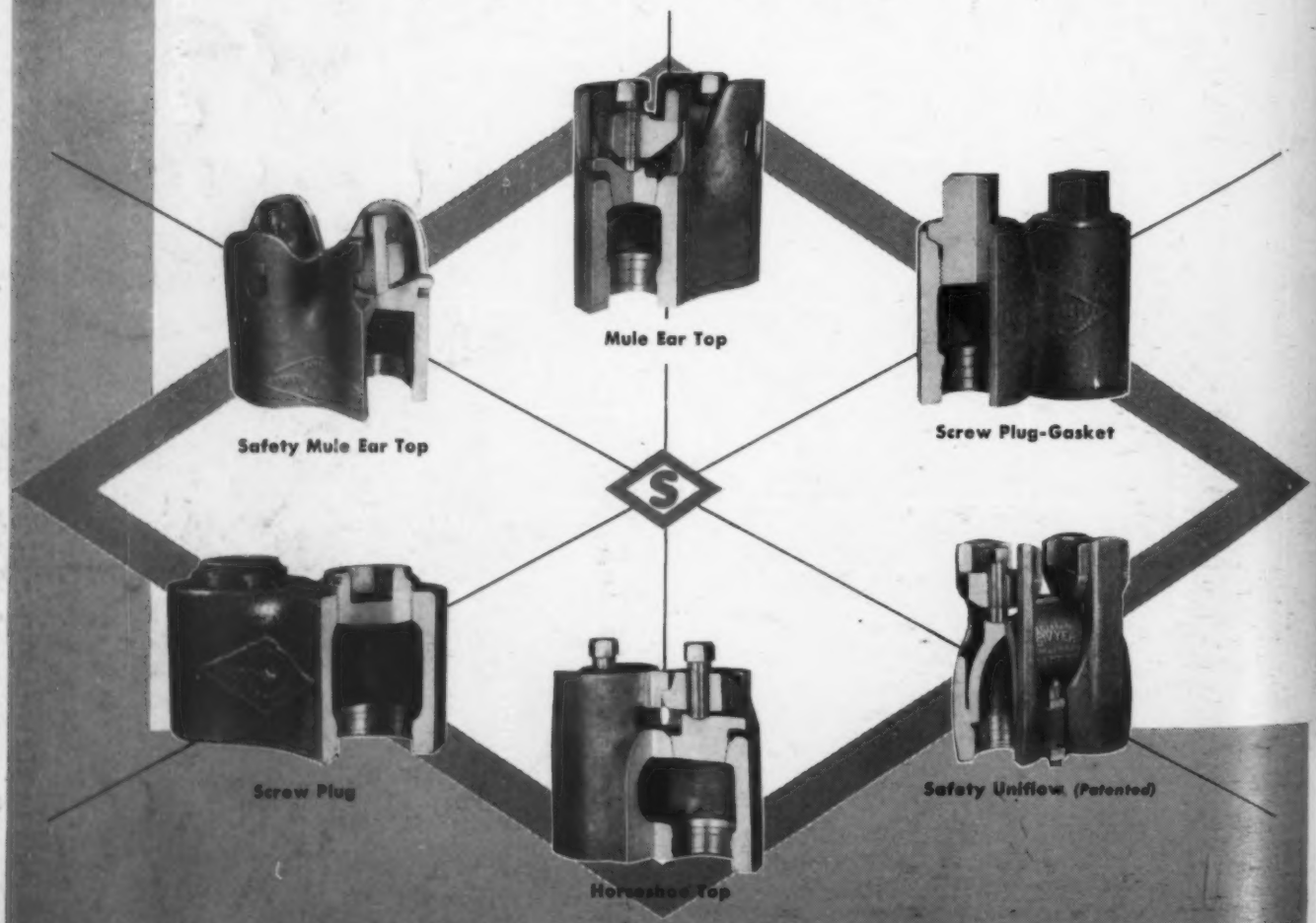

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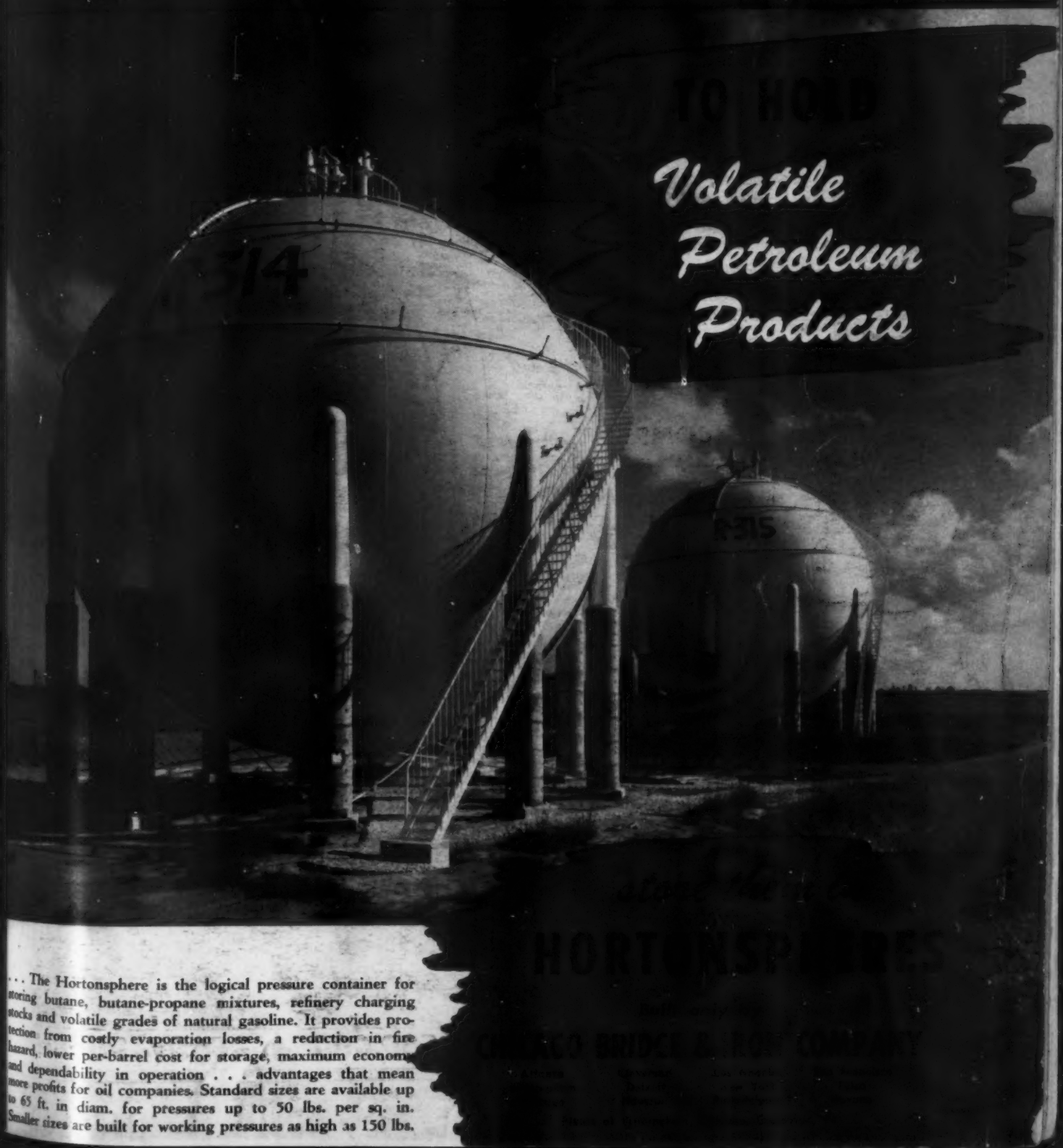

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THE LOOK BOX

WHEN, if and as crude oil is not sufficient, natural gas will supplement the supply, with coal next in line. Industrial developments already underway point to this order of events. Discussion of the relationship involves the prospect of creating the mistaken impression that investigations of gas and coal are admissions that oil is no longer in ample supply. What the events really mean is that industry is planning ahead of actual needs.

Gas and Then Coal

Natural-gas reserves are sufficient for a dozen years of motor-fuel consumption at the current level, admittedly but a moment in the life of a nation. There is coal sufficient for a thousand years, and, in addition, shale reserves are of such magnitude that those who speak of a day when there will be no fuel for the internal-combustion motor are pitifully short in faith and the power of imagination.

Conversion of natural gas into motor fuel, diesel oil and alcohols is at the threshold of commercial status with two plants under construction. Now coal, which will be processed by adaptation of methods used on natural gas, is at the pilot-plant stage with commercial expansion contemplated.

Pittsburgh Consolidated Coal Company will build the pilot plant to process 50 tons of coal daily into 2,400,000 cubic feet of synthesis gas, which can be converted either into motor fuel and other liquid products or into a product similar to natural gas. It is significant that Standard Oil Development Company and Hydrocarbon Research, Inc., are cooperating in this coal program and it is known that other oil companies are pursuing experiments in coal gasification, the first step in its conversion into liquid.

Further development of this program can be appreciated by this part of the statement from Joseph Pursglove, Jr., vice president of the company:

"One single large (commercial) plant of the size being considered could yield a gas of high heat value in quantities equal to the output of the Big Inch and the Little Big Inch pipe lines and create a local source of gasoline, diesel and other fuel oils and a certain amount of alcohols."

Flexibility of such a plant offers this prospect:

"It could be arranged to produce 400,000,000 cubic feet of gas per day in the winter months and reduce this to half that quantity during the summer months when the plant would be set up to produce larger quantities of gasoline. Such a plant would produce an estimated 14,000 barrels of high-octane gasoline per day during the summer season when consumption is highest."

The coal company anticipates building a commercial plant by 1950-51, its cost estimated at $120,000,000. Cost of motor fuel from coal is calculated at 3 to 4 cents per gallon higher than motor

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fuel from natural gas, which is more expensive than motor fuel from crude oil.

Engineering accomplishments and experience will bring reduced costs, an accomplishment common to processing ventures. What is now contemplated is a venture wherein coal will be mined and conversion into carbon monoxide and hydrogen will take place at the surface. These in turn will be converted into liquids or into fuel gas as desired.

Apart from the project of Pittsburgh Consolidated Coal Company is the prospect of coal gasification underground, thus avoiding plant cost and operation for the production of synthesis gas. In Alabama coal is being burned in place to determine the feasibility of gas production. Although this development is far from commercial status its prospects are such that the cost advantage is significant.

Furthermore, if coal can be burned in place to produce a gas for raw material, oil in depleted fields can be so used and experiments are contemplated in this phase of hydrocarbon recovery. Whatever the method, it can be accepted that means will be perfected for going back to get the crude oil that present producing methods will not recover.

It all shapes to assure this nation that worry over sources of liquid energy need not wrinkle the public brow.

Sources Of Supply

IN THEIR paper before the annual meeting of the American Institute of Mining and Metallurgical Engineers in March Robert E. Wilson and J. K. Roberts, Standard Oil Company (Indiana) classed petroleum as an industry due for continued growth for these reasons:

"Although improved design will increase the efficiency of (automotive) engines, this advantage will be taken largely in the form of greater driving flexibility, instead of greater mileage per gallon; and with better roads there will be more driving per car, and at higher speeds. It is therefore expected that the annual consumption of gasoline per car will continue to rise slightly. Commercial vehicles will increase in number at an even faster rate than passenger cars, and annual use of gasoline per commercial vehicle probably will increase by 30 percent by 1970. Diesel equipment will be used more widely, particularly on the railroads and for sea-going vessels. Use of oil heaters for homes is expected to double by 1950, and almost to treble by 1970, by which time more than 20 percent of the one- and two-family homes in the areas using central heat will be heated by oil.

"Increases in these methods of using liquid fuels will be the principal factors in an anticipated 1970 demand for 2,600,000,000 barrels of crude oil or its equivalent—56 percent more than the 1945 demand."

As for sources of supply for this growing demand, the accepted proposition that "the more-easily-found oil has been discovered," leaving natural gas and coal as next in line with the following analysis:

"Known natural-gas reserves would supply all our gasoline requirements for about 12 years, but of course will not be used entirely for this purpose. For an additional 3 or 4 cents per gallon, however, gasoline can be made from coal by a modification of the same (Fischer-Tropsch) process and the U. S. coal reserves are adequate for a thousand years. Gasoline from coal is expected to enter the picture ahead of gasoline from oil shale or tar sands (except possibly for local needs in limited areas) and is expected to become a factor by 1970, earlier if such processes as underground gasification of the coal substantially reduce the costs. Motor fuel from farm products are too expensive to be seriously considered, and with the perfecting of the gasoline-from-coal process are not necessary for the national defense."

Dual Role

FOR agriculture, petroleum is playing the dual role of life for crops and death for weeds.

In the one it finds its way into fertilizers. Natural gas by conversion gets into ammonia, a basic chemical for growth aid. As ethylene it speeds the ripening of fruit. And there are scores of other adaptations, so many in fact that the industry is setting up its laboratories to expend its sales in the agricultural field.

Then as a destructive fluid, solvents take their toll of weeds, while giving no harm to certain food crops.

Since the American farm is rapidly shifting to motor-driven equipment, the role of oil is in reality triple.

Brakes Dragging

IN THE annual report of Cities Service Oil Company, W. Alton Jones, president, pointed out that extension of petro-chemical processing faces the barrier of taxation, saying:

"The development of these chemical by-products and markets will require enormous expenditures of money. The money can come only out of earnings or from new capital investments. Heavy taxes impair both these sources. A discriminatory tax affects the natural progress of the industry and, more importantly, deprives the public of the benefits of new products and services essential to an expanding economy."

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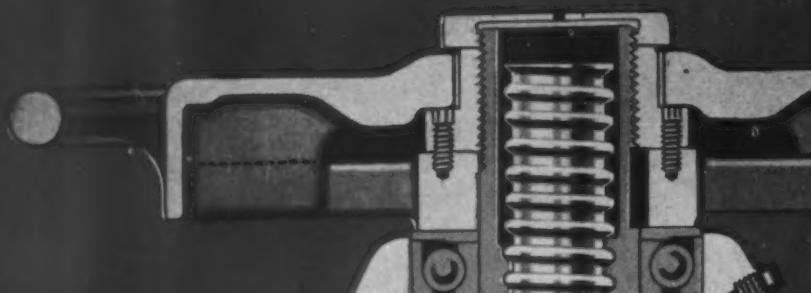

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Edward developed the ingeniously simple patented EValthrust yoke bushing to facilitate the closing of big valves against tremendous pressures. With its companion unit, the patented Edward Impactor handwheel, the EValthrust yoke bushing gives maximum closing torque in minimum space.

In thousands of important installations throughout the country, EValthrust equipped Edward valves are busy, convincing proof that yoke bushing failures are wholly avoidable; that even the largest valves can be closed easily and tightly, though space is restricted and pressures and temperatures are extreme.

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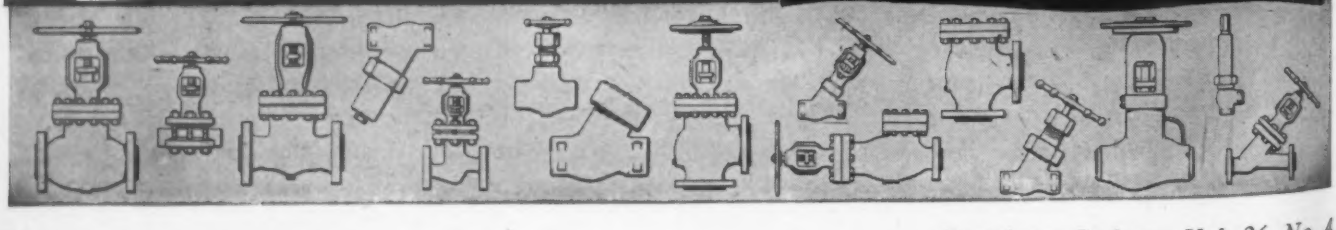

Utilization of Natural Gas For Chemical Products

HAROLD M. SMITH
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and
W. C. HOLLIMAN
Research Chemist (Petroleum)

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THIS paper was released in March, 1947, as Information Circular 7347 of the United States Bureau of Mines. In addition to the complete manuscript and pertinent bibliography, the circular offers 218 selected references, many of which were given in the extensive bibliography of Weil and Lane in their four recent articles "The Synthine Process" in Petroleum Refiner. For that reason these references are not reproduced here.

Although the chemicals either being made or known to be possible from natural gas make an impressive list, the extent to which this raw material may be expanded is really more impressive. Its adaptability and availability have had this influence, as stated by the authors:

"Many chemicals not previously procurable commercially have been made from natural-gas hydrocarbons and in many instances they have filled needs that had not been met by other known substances."

AMONG the more important raw materials now utilized for the commercial production of synthetic organic chemicals is natural gas. Natural gas has been used as a fuel and source of energy almost from its discovery, but its potentialities as a chemical raw material were not realized until recently.

Research in the use of natural gas for chemical synthesis received great impetus from the discovery by Brooks and Humphrey[1] in 1918 of alcohols in the diluted "acid oil" obtained by sulfuric acid treatment of cracked petroleum distillates. Experimental data soon showed that hydrocarbons from natural gas could be converted to these same products. Laboratory methods for chlorination of natural-gas hydrocarbons, particularly of methane for the production of methyl chloride, methylene chloride, carbon tetrachloride, and chloroform were known before that time but were not developed commercially. Methods for obtaining alcohol by oxidation of hydrocarbons derived from natural gas also were known before 1918 but had not received commercial consideration. Since 1918, and particularly during the past 15 years, the work of chemists and chemical manufacturers throughout the world has resulted in an accumulation of data regarding the reactivity of natural-gas hydrocarbons, and related hydrocarbons found in refinery gases or made synthetically. These data have been used to develop methods for converting hydrocarbons into chemical products and thus form the basis for a large and growing industry.

Wartime demands for chemicals during 1939-1945 gave impetus to this development, especially to the application of engineering technology to methods already known in the laboratory but which had not been developed commercially. The production of plastics as substitutes for rubber and silk; of ammonia, glycerine, toluene, and nitroparaffins for use in explosives; of synthetic rubber; and of aviation gasoline are examples of this technologic growth. The demand for these products required new sources of raw materials, and as a result many processes were developed and plants constructed to utilize natural gas and other petroleum hydrocarbons. With the cessation of hostilities, the conversion of these plants and processes to peacetime activities has been rapid. Additional postwar construction of plants for chemical utilization of petroleum gases indicates that the growth of this phase of the petroleum industry will continue.

The quantity of natural gas that is, or that under present demands can be, utilized in the production of chemicals is not great in comparison with the amount used as fuel. Some data showing present consumption of natural gas for several purposes are presented and discussed with Chart 2. Generally, an organic chemical plant can be made from any one of several starting materials as well as by several routes from a single substance. This is illustrated by and discussed with Chart 5. This versatility of hydrocarbon-conversion processes is important, as ordinarily it may be assumed that a substance will not be made synthetically from naturally-occurring raw materials if it can be obtained in sufficient quantities as a by-product from an active commercial process. The cheapness and availability of natural gas, however, has caused unusual consideration to be given to its chemical utilization. Many chemicals not previously procurable commercially have been made from natural-gas hydrocarbons, and in many instances they have filled needs that had not been met by other known substances.

The production, from natural gas, of aviation gasoline and of high-antiknock blending agents for enrichment of motor fuels is not strictly a "utilization for chemical products." Many of these gasoline components, however, are relatively pure chemicals, and the processes employed in their manufacture are applicable to the manufacture of products intended for chemical rather than energy uses. Investigations of production of motor fuels from natural gas and refinery-by-product gases, which were begun actively about 1928, resulted in the discovery of many processes. These investigations also disclosed additional methods and technology for production of pure chem-

icals and chemical raw materials from natural-gas hydrocarbons. A report giving the results of some of the early investigations of this problem by the Bureau of Mines were published in October, 1931.[2] In that report, previous and concurrent work by other investigators is cited.

An information circular concerning the utilization of natural gas for chemical products was published by the Bureau in 1930[3] and a revision was issued in 1940.[4] The present paper is a revision of the 1940 publication. Its purpose is to show in graphic form the operating conditions are so varied that no simple explanation seems possible. Actually, few of the reactions are as simple as the charts may indicate. The subject of catalysts alone is so complex that it cannot be presented adequately in a report such as this. The nontechnical reader is interested mainly in the general picture which is adequately shown by the charts.

Chart 1

Natural gas is a mixture of gaseous hydrocarbons consisting predominantly of those containing not

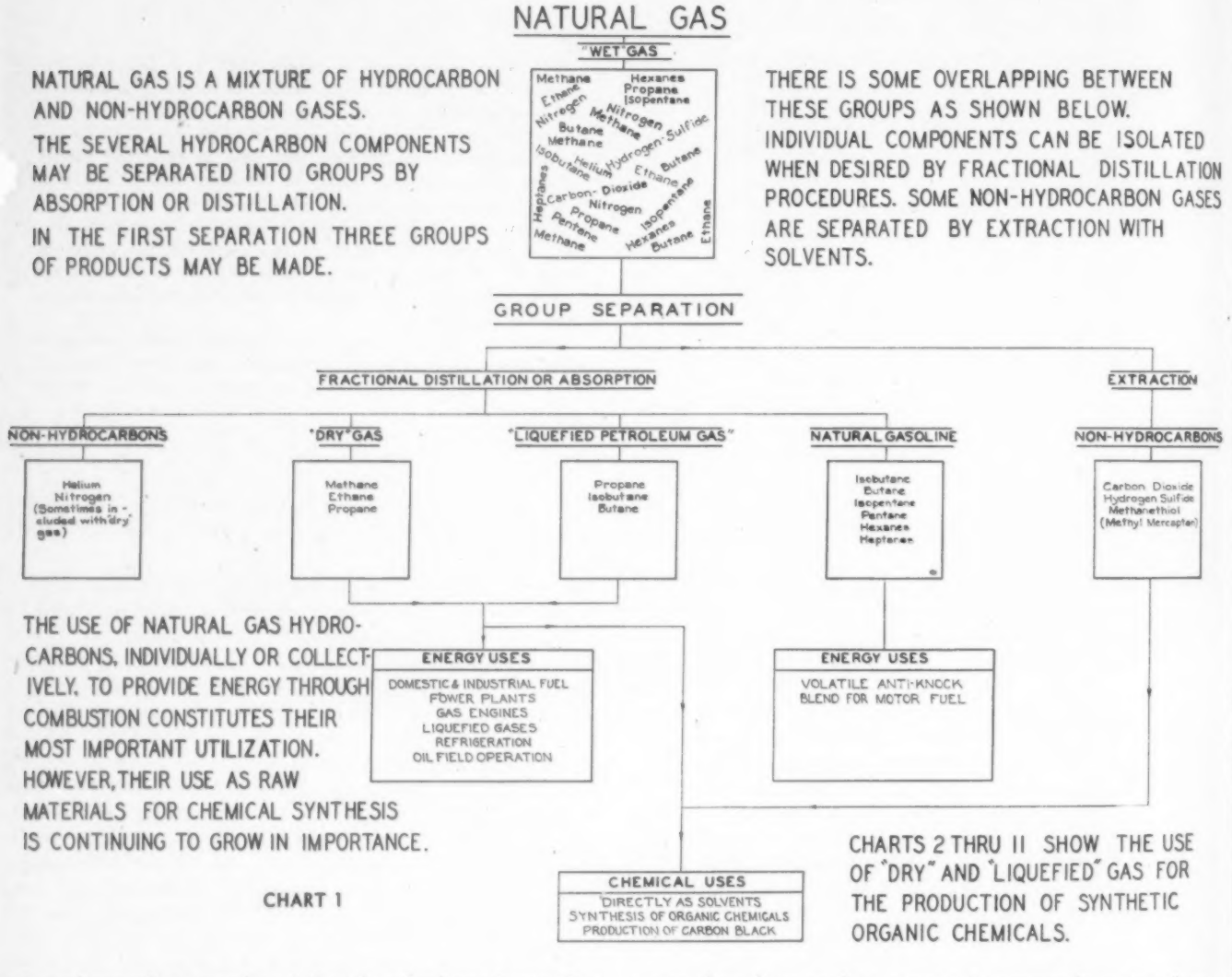

CHART 1

general reactions used and the chemical products that may be obtained when natural gas is used as a raw material. The number of products presently obtainable from natural gas is very large, and the field is continuing to develop, so that it is possible that some products that are now important (or soon may be so) have been omitted, but every effort has been made to make this publication as inclusive as available information permits. The products listed may be produced from natural gas from wells or natural-gasoline plants, but ordinarily natural gas would not be used where refinery-process gases or other raw materials are available that will yield the desired product in fewer steps and more cheaply. Some statistics relating to the economics and growth of the industry are shown in the first charts, and the principal processes and their products are shown in the others.

It would be desirable to include brief descriptions of some of the processes, but choices of methods and more than four carbon atoms. It occurs in natural reservoirs separately as gas or may be found with or in solution in nongaseous hydrocarbons. The composition of natural gas may differ from field to field and also with production methods. Methane is the hydrocarbon usually present in greatest volume, with ethane, propane, and heavier hydrocarbons successively next in order. The composition of a "wet" gas typical of much of that produced lies within the following limits in volume-percent: Methane, 80-90 percent; ethane, 5-10 percent; propane, 3-5 percent; isobutane and butane, 1-2 percent; pentanes and heavier hydrocarbons, 1-2 percent. Nonhydrocarbon components, including nitrogen, hydrogen sulfide, traces of organic sulfur compounds, carbon dioxide, and helium, may occur with the natural gas. Certain of these may be extracted from natural gas either as impurities or for individual utilization. (Chart 12.)

Chart 1 shows the general composition and major

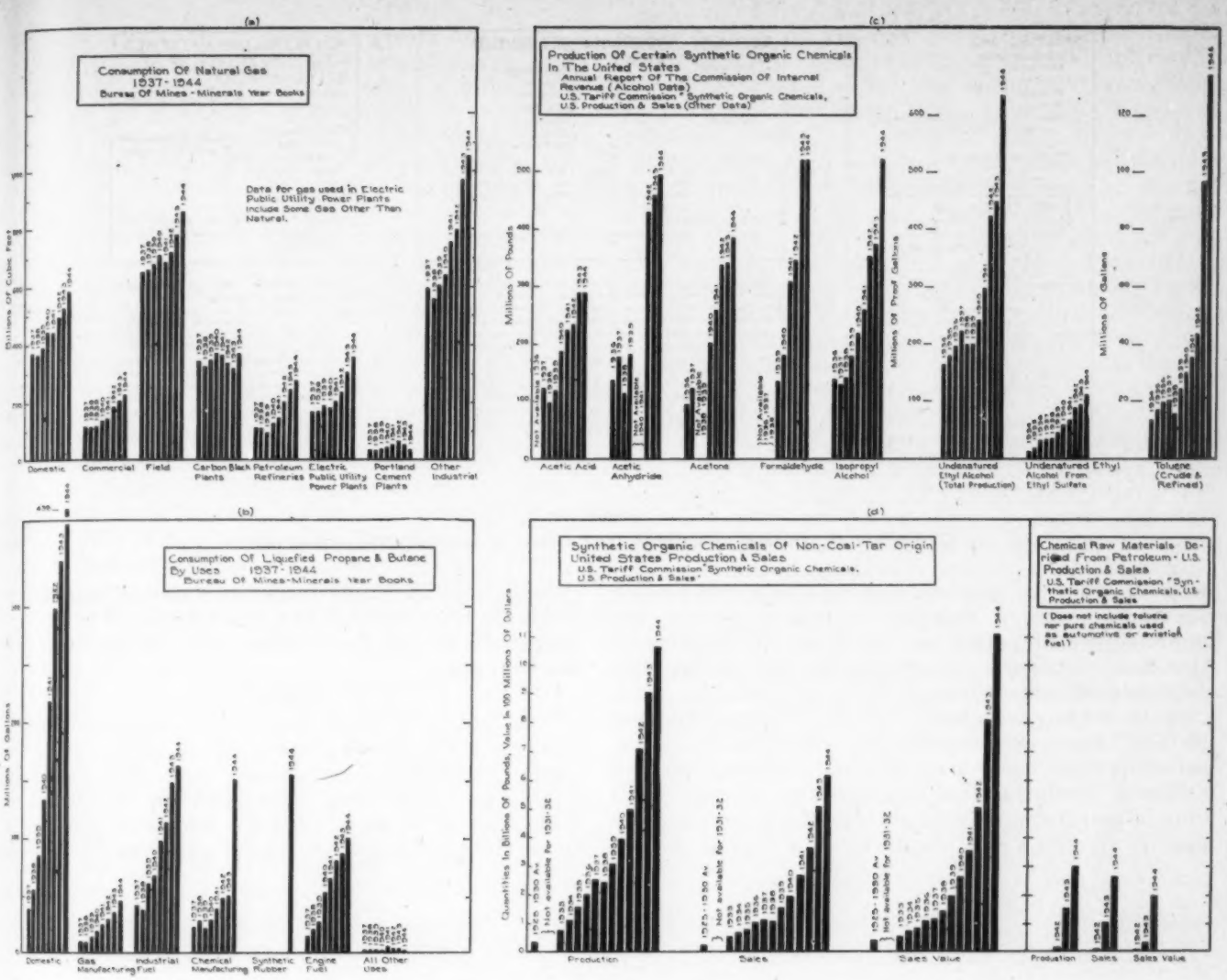
(a)
Consumption Of Natural Gas
1937-1944
Bureau Of Mines - Minerals Year Books
Data for gas used in Electric Public Utility Power Plants include Some Gas Other Than Natural.
Billions Of Cubic Feet
Domestic
Commercial
Field
Carbon Black Plants
Petroleum Refineries
Electric Public Utility Power Plants
Portland Cement Plants
Other Industrial
(b)
Consumption Of Liquefied Propane & Butane By Uses 1937-1944
Bureau Of Mines-Minerals Year Books
Millions Of Gallons
Domestic
Gas Manufacturing
Industrial Fuel
Chemical Manufacturing
Synthetic Rubber
Engine Fuel
All Other Uses
(c)
Production Of Certain Synthetic Organic Chemicals In The United States
Annual Report Of The Commission Of Internal Revenue (Alcohol Data)
U.S. Tariff Commission "Synthetic Organic Chemicals, U.S. Production & Sales (Other Data)
Millions Of Pounds
Millions Of Proof Gallons
Millions Of Gallons
Acetic Acid
Acetic Anhydride
Acetone
Formaldehyde
Isopropyl Alcohol
Undenatured Ethyl Alcohol (Total Production)
Undenatured Alcohol From Ethyl Sulfate
Ethyl Toluene (Crude & Refined)
(d)
Synthetic Organic Chemicals Of Non-Coal-Tar Origin United States Production & Sales
U.S. Tariff Commission "Synthetic Organic Chemicals, U.S. Production & Sales"
Quantities In Billions Of Pounds, Value In 100 Millions Of Dollars
Production
Sales
Sales Value
Chemical Raw Materials Derived From Petroleum - U.S. Production & Sales
U.S. Tariff Commission "Synthetic Organic Chemicals, U.S. Production & Sales"
(Does not include toluene nor pure chemicals used as automotive or aviation fuel)
Production
Sales
Sales Value

CHART 2

uses of natural gas. The gaseous mixture or "wet" gas produced at the wellhead, as shown in the top square of the chart, usually is separated in the field in absorption or natural-gasoline plants. Nonhydrocarbons are removed under certain conditions, and the hydrocarbons are separated into "dry gas," "liquefied petroleum gas," and natural gasoline or condensate. ("Dry" gas occurs as such in some fields. When gas is produced with crude oil, a rough field separation gives a wet gas.) Such separations are not ordinarily complete, and there is some overlapping between the groups. Further group separation or separation into individual components is accomplished when desired by fractional distillation, or in special cases by extractive distillation, azeotropic distillation, superfractionation, or other extractive methods.

Data regarding uses of the several constituents are shown in Chart 2. Charts 7 to 11, which show chemical products, are concerned primarily with "dry" and "liquefied petroleum gases" hydrocarbons—methane, ethane, propane, isobutane, and n-butane.

Chart 2

Some data regarding the consumption and use of natural and liquefied petroleum gas are shown graphically in Charts 2a and 2b. Unfortunately, figures for the quantity of natural gas used in the manufacture of synthetic organic chemicals are not available separately but are included under "other industrial" uses. Data on the use of liquefied petroleum gases in the manufacture of synthetic-rubber components were available for the first time in 1944. Data concerning the use of liquefied petroleum gas in the production of aviation gasoline and other motor-fuel components are not included. Chart 2c shows annual synthetic production from all sources of certain non-coal-tar chemicals since 1936. Chart 2d shows the increase in total production and sales of non-coal-tar synthetic organic chemicals from 1925 to 1944 and production and sales of chemicals raw materials derived from petroleum from 1942 to 1944.

Statistics showing the portion of this production using natural gas as the raw material are not available.

Chart 3

Chart 3 shows the general composition of crude petroleum and of refinery-processed petroleum and also lists some non-petroleum materials suitable for hydrocarbon synthesis. Petroleum is an extremely complex mixture of hydrocarbons and other substances that is found in nature in the solid, liquid, and gaseous states. Natural gas is a form of petroleum that consists of hydrocarbons and other substances (Chart 1) that exists in the gaseous state under ordinary conditions of temperature and pressure at the earth's surface. The natural-gas hydrocarbons frequently occur in solution with crude oil. Hydrocarbons identical with those in natural gas, together

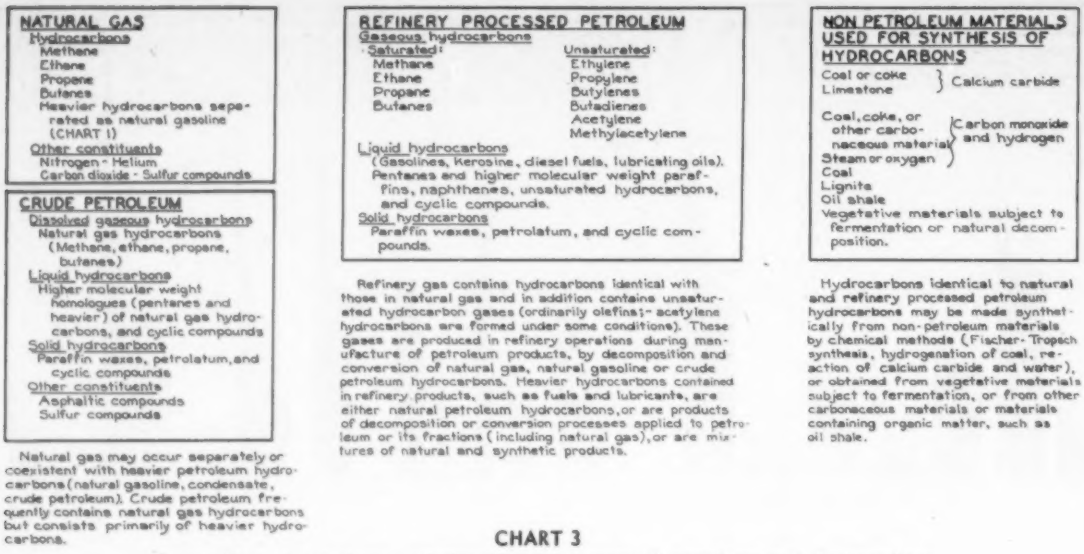
NATURAL GAS
Hydrocarbons
Methane
Ethane
Propane
Butanes
Heavier hydrocarbons separated as natural gasoline (CHART 1)
Other constituents
Nitrogen · Helium
Carbon dioxide · Sulfur compounds
CRUDE PETROLEUM
Dissolved gaseous hydrocarbons
Natural gas hydrocarbons (Methane, ethane, propane, butanes)
Liquid hydrocarbons
Higher molecular weight homologues (pentanes and heavier) of natural gas hydrocarbons, and cyclic compounds
Solid hydrocarbons
Paraffin waxes, petrolatum, and cyclic compounds
Other constituents
Asphaltic compounds
Sulfur compounds
Natural gas may occur separately or coexistent with heavier petroleum hydrocarbons (natural gasoline, condensate, crude petroleum). Crude petroleum frequently contains natural gas hydrocarbons but consists primarily of heavier hydrocarbons.
REFINERY PROCESSED PETROLEUM
Gaseous hydrocarbons
Saturated:
Methane
Ethane
Propane
Butanes
Unsaturated:
Ethylene
Propylene
Butylenes
Butadienes
Acetylene
Methylacetylene
Liquid hydrocarbons
(Gasolines, Kerosine, diesel fuels, lubricating oils). Pentanes and higher molecular weight paraffins, naphthenes, unsaturated hydrocarbons, and cyclic compounds.
Solid hydrocarbons
Paraffin waxes, petrolatum, and cyclic compounds.
Refinery gas contains hydrocarbons identical with those in natural gas and in addition contains unsaturated hydrocarbon gases (ordinarily olefins - acetylene hydrocarbons are formed under some conditions). These gases are produced in refinery operations during manufacture of petroleum products, by decomposition and conversion of natural gas, natural gasoline or crude petroleum hydrocarbons. Heavier hydrocarbons contained in refinery products, such as fuels and lubricants, are either natural petroleum hydrocarbons, or are products of decomposition or conversion processes applied to petroleum or its fractions (including natural gas), or are mixtures of natural and synthetic products.
NON PETROLEUM MATERIALS USED FOR SYNTHESIS OF HYDROCARBONS
Coal or coke
Limestone
} Calcium carbide
Coal, coke, or other carbonaceous material
Steam or oxygen
} Carbon monoxide and hydrogen
Coal
Lignite
Oil shale
Vegetative materials subject to fermentation or natural decomposition.
Hydrocarbons identical to natural and refinery processed petroleum hydrocarbons may be made synthetically from non-petroleum materials by chemical methods (Fischer-Tropsch synthesis, hydrogenation of coal, reaction of calcium carbide and water), or obtained from vegetative materials subject to fermentation, or from other carbonaceous materials or materials containing organic matter, such as oil shale.

CHART 3

Composition of petroleum—relationship and sources of petroleum-type hydrocarbons.

with unsaturated gaseous hydrocarbons, are formed as by-products in refinery operations during the manufacture of petroleum products, by decomposition and conversion of natural-gas, natural-gasoline, or crude-oil hydrocarbons. Such hydrocarbons also may be made synthetically from non-petroleum materials. These gaseous products, regardless of source, are important raw materials or intermediates for chemical products. The fact that the gaseous products of petroleum may be obtained by processing the heavier fractions and, similarly, that heavier hydrocarbons may be made from the gases is significant. Methods whereby such interconversion of petroleum hydrocarbons can be accomplished are presented in the next chart.

Chart 4

Raw materials other than petroleum hydrocarbons were used for the first commercial syntheses of organic chemicals. The processes, however, usually involved as a first step hydrogenation of carbon or formation of hydrogen-carbon products similar to those already present in natural gas or easily derived

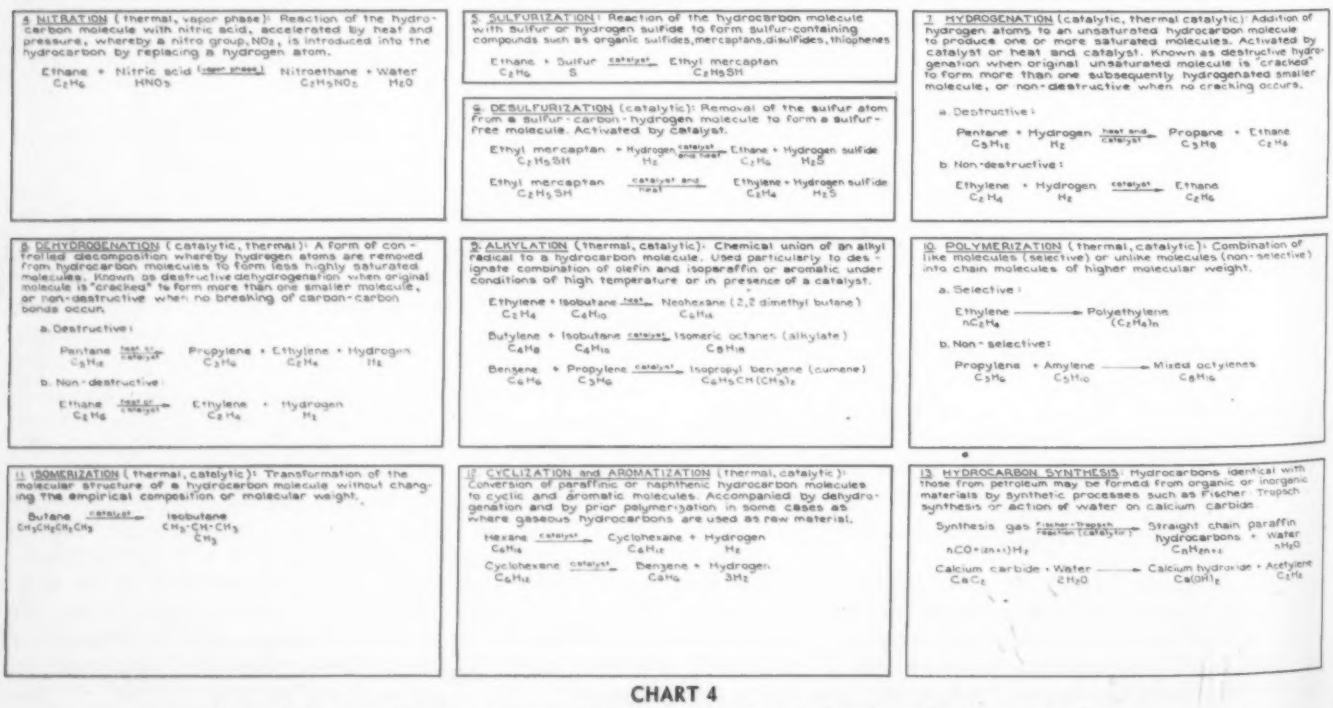
1. DECOMPOSITION (thermal, catalytic, electric): The splitting of the hydrocarbon molecule into smaller molecules or into carbon and hydrogen by heat alone (pyrolysis), with the aid of catalysts, or by electric discharge. Generally accompanied, especially in pyrolysis, by recombination of some of the products into new compounds.
Ethane → Hydrogen + Ethylene
Ethane → Hydrogen + Acetylene
Ethane → Hydrogen + Carbon
2. OXIDATION (thermal, catalytic): Reaction of the hydrocarbon molecule with oxygen, air or oxygen-containing compounds, activated by heat or catalyst, whereby oxygen is introduced into the hydrocarbon molecule, or the molecule is changed to carbon monoxide and hydrogen, carbon dioxide and hydrogen, or finally to carbon dioxide and water.
Ethane + Oxygen → Ethyl alcohol
Ethane + Water (Steam) → Hydrogen + Carbon dioxide
Ethane → Water + Carbon dioxide
3. HALOGENATION (thermal, catalytic, photolytic): Reaction of the hydrocarbon molecule with a halogen (fluorine, chlorine, bromine, iodine), activated by heat, light, or catalyst, whereby one or more halogen atoms replace an equivalent number of hydrogen atoms in the hydrocarbon molecule.
Ethane + Chlorine → Ethyl chloride + Hydrogen chloride
4. NITRATION (thermal, vapor phase): Reaction of the hydrocarbon molecule with nitric acid, accelerated by heat and pressure, whereby a nitro group, NO_2, is introduced into the hydrocarbon by replacing a hydrogen atom.
Ethane + Nitric acid (vapor phase) → Nitroethane + Water
5. SULFURIZATION: Reaction of the hydrocarbon molecule with sulfur or hydrogen sulfide to form sulfur-containing compounds such as organic sulfides, mercaptans, disulfides, thiophenes
Ethane + Sulfur → Ethyl mercaptan
6. DESULFURIZATION (catalytic): Removal of the sulfur atom from a sulfur-carbon-hydrogen molecule to form a sulfur-free molecule. Activated by catalyst.
Ethyl mercaptan + Hydrogen → Ethane + Hydrogen sulfide
Ethyl mercaptan → Ethylene + Hydrogen sulfide
7. HYDROGENATION (catalytic, thermal catalytic): Addition of hydrogen atoms to an unsaturated hydrocarbon molecule to produce one or more saturated molecules. Activated by catalyst or heat and catalyst. Known as destructive hydrogenation when original unsaturated molecule is "cracked" to form more than one subsequently hydrogenated smaller molecule, or non-destructive when no cracking occurs.
a. Destructive:
Pentene + Hydrogen → Propane + Ethane
b. Non-destructive:
Ethylene + Hydrogen → Ethane
8. DEHYDROGENATION (catalytic, thermal): A form of controlled decomposition whereby hydrogen atoms are removed from hydrocarbon molecules to form less highly saturated molecules. Known as destructive dehydrogenation when original molecule is "cracked" to form more than one smaller molecule, or non-destructive when no breaking of carbon-carbon bonds occurs.
a. Destructive:
Pentane → Propylene + Ethylene + Hydrogen
b. Non-destructive:
Ethane → Ethylene + Hydrogen
9. ALKYLATION (thermal, catalytic): Chemical union of an alkyl radical to a hydrocarbon molecule. Used particularly to designate combination of olefin and isoparaffin or aromatic under conditions of high temperature or in presence of a catalyst.
Ethylene + Isobutane → Neohexane (2,2 dimethyl butane)
Butylene + Isobutane → Isomeric octanes (alkylate)
Benzene + Propylene → Isopropyl benzene (cumene)
10. POLYMERIZATION (thermal, catalytic): Combination of like molecules (selective) or unlike molecules (non-selective) into chain molecules of higher molecular weight.
a. Selective:
Ethylene → Polyethylene
b. Non-selective:
Propylene + Amylene → Mixed octylenes
11. ISOMERIZATION (thermal, catalytic): Transformation of the molecular structure of a hydrocarbon molecule without changing the empirical composition or molecular weight.
Butane → Isobutane
12. CYCLIZATION and AROMATIZATION (thermal, catalytic): Conversion of paraffinic or naphthenic hydrocarbon molecules to cyclic and aromatic molecules. Accompanied by dehydrogenation and by prior polymerization in some cases as where gaseous hydrocarbons are used as raw material.
Hexane → Cyclohexane + Hydrogen
Cyclohexane → Benzene + Hydrogen
13. HYDROCARBON SYNTHESIS: Hydrocarbons identical with those from petroleum may be formed from organic or inorganic materials by synthetic processes such as Fischer Tropsch synthesis or action of water on calcium carbide.
Synthesis gas → Straight chain paraffin hydrocarbons + Water
Calcium carbide + Water → Calcium hydroxide + Acetylene

CHART 4

Methods for conversion of petroleum hydrocarbons, directly or indirectly applicable to natural gas.

from petroleum. It was soon recognized that petroleum and natural-gas hydrocarbons offer raw materials suitable in their original form, or after rearrangement to new molecular structure, for use in synthesis of chemical products. Further steps in the production of synthetic organic chemicals involve the addition or substitution of other elements such as oxygen, chlorine, nitrogen, or sulfur to the hydrocarbon structure. Typical methods for rearranging the hydrocarbon molecule and for converting it into other products are shown and defined in Chart 4.

Probably the most important method used for converting petroleum hydrocarbons to other compounds is decomposition effected by pyrolysis, catalysis, or electric discharge. Decomposition as used here also includes, in many instances, subsequent recombination of primary products into new compounds. Dehydrogenation and hydrogenation, alkylation, polymerization, isomerization, desulfurization, and cyclization and aromatization all may be considered as special cases of controlled decomposition or decomposition and recombination. Some metohds of oxidation, halogenation, nitration, sulfurization, and amination involve preliminary decomposition of saturated hydrocarbons to olefins and recombination with the added elements.

Decomposition, oxidation, halogenation, nitration, and sulfurization are further discussed with the charts showing products obtained by use of the respective methods. Many of the products shown on these charts are the result of a second series of reactions or conversions using the primary products as starting materials. Some typical examples of such reactions are shown in Chart 4, and the following deserve special mention: Alkylation and hydrogenation have been of especial importance in the production of high-antiknock blending agents for aviation gasoline; isomerization has been used extensively to convert n-butane or n-pentane to isobutane or isopentane for use in alkylation and in aviation gasoline. Selective and non-selective polymerization is used in the manufacture of intermediates for plastics and synthetic rubbers as well as in making motor fuels and lubricants; desulfurization is often a necessary process in the production of motor fuels; dehydrogenation (and sometimes polymerization) together with cyclization and aromatization is used in the production of aromatics and cyclic compounds. The development of processes using these conversion methods received much consideration before and during the recent war, and processes developed now are being adapted to peacetime needs.

It has been mentioned already that coal, petroleum, agricultural products, and other carbonaceous materials may be used interchangeably as raw materials for hydrocarbon synthesis and manufacture of chemicals. Hydrocarbon synthesis and production of chemicals from materials other than petroleum has been used in Germany and in our own country as a wartime measure. Liquid fuels, lubricants, materials for synthetic rubber, and a large number of organic chemicals were produced in Germany from coal (or in some cases natural gas).

Among the processes using coal or coke as raw material are the production of calcium carbide for conversion into acetylene, the hydrogenation of coal to produce gaseous and liquid hydrocarbons, and the Fischer-Tropsch synthesis, which involves a prior oxidation of coal or other carbonaceous material with oxygen or steam. Natural gas may be used in this process instead of coal, and this application has received much interest recently. It is discussed further with the chart showing products obtained by oxidation (Chart 8). Similarly, acetylene is produced also by decomposition of natural gas or gas from coal hydrogenation and is discussed further with Chart 7.

Chart 5

Chart 5 shows diagrammatically the production of butadiene by several possible methods using some of the possible raw materials. The chart illustrates the diverse routes by which a desired organic chemical may be obtained. Economic considerations involved usually determine the choice of raw materials and route. Butadiene for synthetic rubber was produced from coal in Germany during the war, whereas in America it was made on an extensive scale from ethyl alcohol, which was obtained from agricultural sources or from petroleum; also, directly from petroleum hydrocarbons, chiefly from butylenes and butanes. The choice of starting material in each instance was determined by the availability and need for the several types of materials. In Germany, coal was the initial

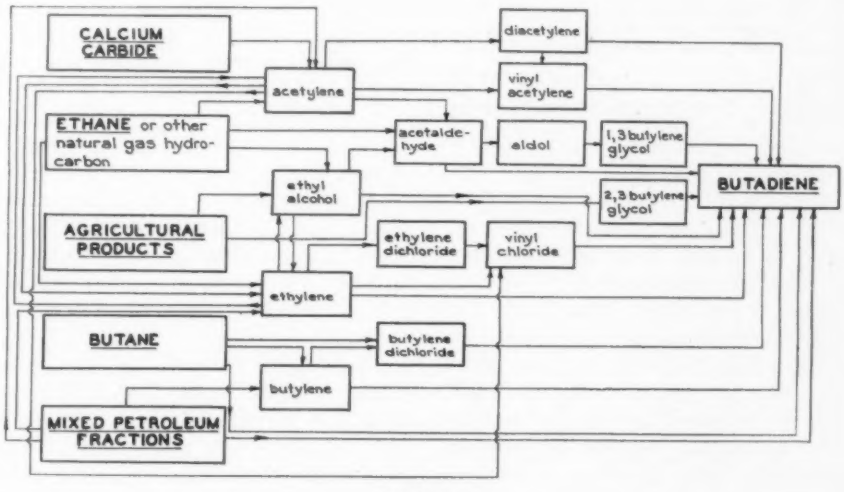

CHART 5

Several possible methods by which butadiene may be produced.

raw material, but actual butadiene production was mainly from by-product gases obtained in the hydrogenation of coal. These gases were converted into acetylene and ethylene by means of the electric arc, and the acetylene was converted to acetaldehyde and then to butadiene by the aldol route.

Chart 6

Chart 6 should be used for reference in studying Charts 7 to 12, as it indicates the significance of the several "boxes," brackets, and types of lettering used in them. In Chart 6 the terms used have the following meanings:

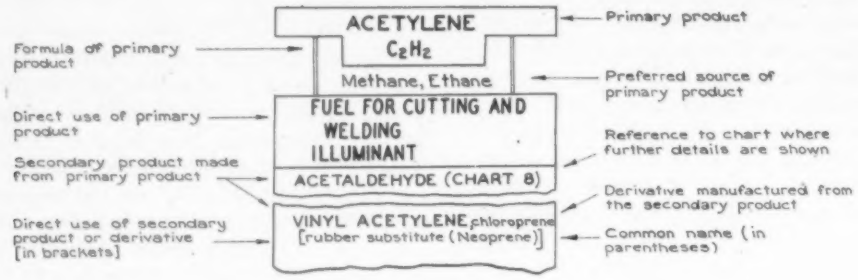
ACETYLENE
C_2H_2
Methane, Ethane
FUEL FOR CUTTING AND WELDING ILLUMINANT
ACETALDEHYDE (CHART 8)
VINYL ACETYLENE, chloroprene [rubber substitute (Neoprene)]
Formula of primary product
Primary product
Preferred source of primary product
Direct use of primary product
Reference to chart where further details are shown
Secondary product made from primary product
Derivative manufactured from the secondary product
Direct use of secondary product or derivative [in brackets]
Common name (in parentheses)

CHART 6

Primary product.—A product obtained directly by the application of the basic method of conversion indicated in the title of the individual chart.

Preferred source.—Not necessarily the only source, but the material reacting most easily or with the best yields relative to other natural-gas hydrocarbons (see remarks under "Charts 7 to 11—General").

Direct use.—A use (a) in which no chemical change occurs in the substance, as when it is used as a solvent or anesthetic, or (b) when the substance is completely destroyed, as when used for fuel. The direct uses of secondary products are indicated in brackets.

Secondary product.—A product made from the primary product. common names of secondary products and their derivatives are indicated in parenthesis. Direct uses are shown in brackets.

Chart 7 to 11 (General)

In preparing Charts 7 to 11, products shown as derivatives of a particular hydrocarbon are not necessarily produced in commercial practice from that hydrocarbon or from a hydrocarbon as the original raw material. They are included, however, as they could be produced as shown if desirable to do so.

Examples of compounds of this type are the "Freons," which are shown as secondary products obtainable from natural-gas hydrocarbons by halogenation. These refrigerants are produced commercially, according to best available information, from carbon tetrachloride or other compounds derived from non-hydrocarbon raw materials. Data on raw materials actually used in commercial production of synthetic chemicals are not revealed by many manufacturers.

Space does not permit naming all possible derivatives or secondary products. For example, one company lists 85 organic chemicals, of which they say: "In producing all these chemicals, olefins derived by processing natural gas are used either directly or indirectly as raw material." Disclosure of scientific information from enemy sources since the recent war show that German technicians produced 37 or more chemical products from acetylene. An American company manufacturing nitration products from natural-gas hydrocarbons lists 54 derivatives as an incomplete list of those that can be made available. A handbook showing uses and applications of chemicals shows butyl alcohol as a reagent in making 132 different compounds. These data show the impracticability of listing all products and indicate the possibilities of chemical production with natural gas as a raw material. The use of heavier petroleum hydrocarbons, which are nongaseous under ordinary conditions, as initial materials for chemical production also should be considered.

Chart 7

The decomposition of natural-gas hydrocarbons into smaller reactive molecules (primary products), which may recombine during the operation to form larger hydrocarbon molecules, either aliphatic or cyclic, or which may be separated and utilized as intermediate products in the production of fuels and chemicals, is perhaps the most important of the processes for conversion of these hydrocarbons. These primary products usually are more susceptible to chemical manipulation than the original molecules.

Thermal (pyrolytic), thermal catalytic, and electrical methods are applied successfully to the decomposition of petroleum and natural-gas hydrocarbons. The first two of these have found greatest application. The electric arc has been reported to have been used successfully for decomposition of natural-gas hydrocarbons; it was used in Germany during the war for the production of acetylene from natural gas of from by-product gas from coal hydrogenation.

Primary products from natural-gas decomposition shown in Chart 7 do not include aliphatic hydrocarbons containing more than four carbon atoms or the less common homologues of acetylene or butadiene that may be formed. Because of the attention given to some of these primary products in recent years, more individual mention is deserved than can be given in the charts.

Hydrogen is produced from natural gas by thermal decomposition with concurrent production of carbon black. Another process for production of hydrogen from natural gas (by reaction with steam) is shown in Chart 8 and discussed in the accompanying discussion. Both of these methods are used extensively for the production of hydrogen for use in the hydrogenation of oils, in the manufacture of ammonia, and for other uses.

The production and chemistry of acetylene received much attention long before methods were evolved for its production from natural-gas hydrocarbons. Although acetylene presents some mechanical difficulty in handling because of its tendency to explode under pressure, its use in chemical synthesis has attained a position of importance in American synthetic processes. It is used as the basis for the manufacture of a number of commercial products and was used widely in Germany for production of chemicals needed throughout the war. Ordinarily, acetylene is produced for commercial use from non-hydro-

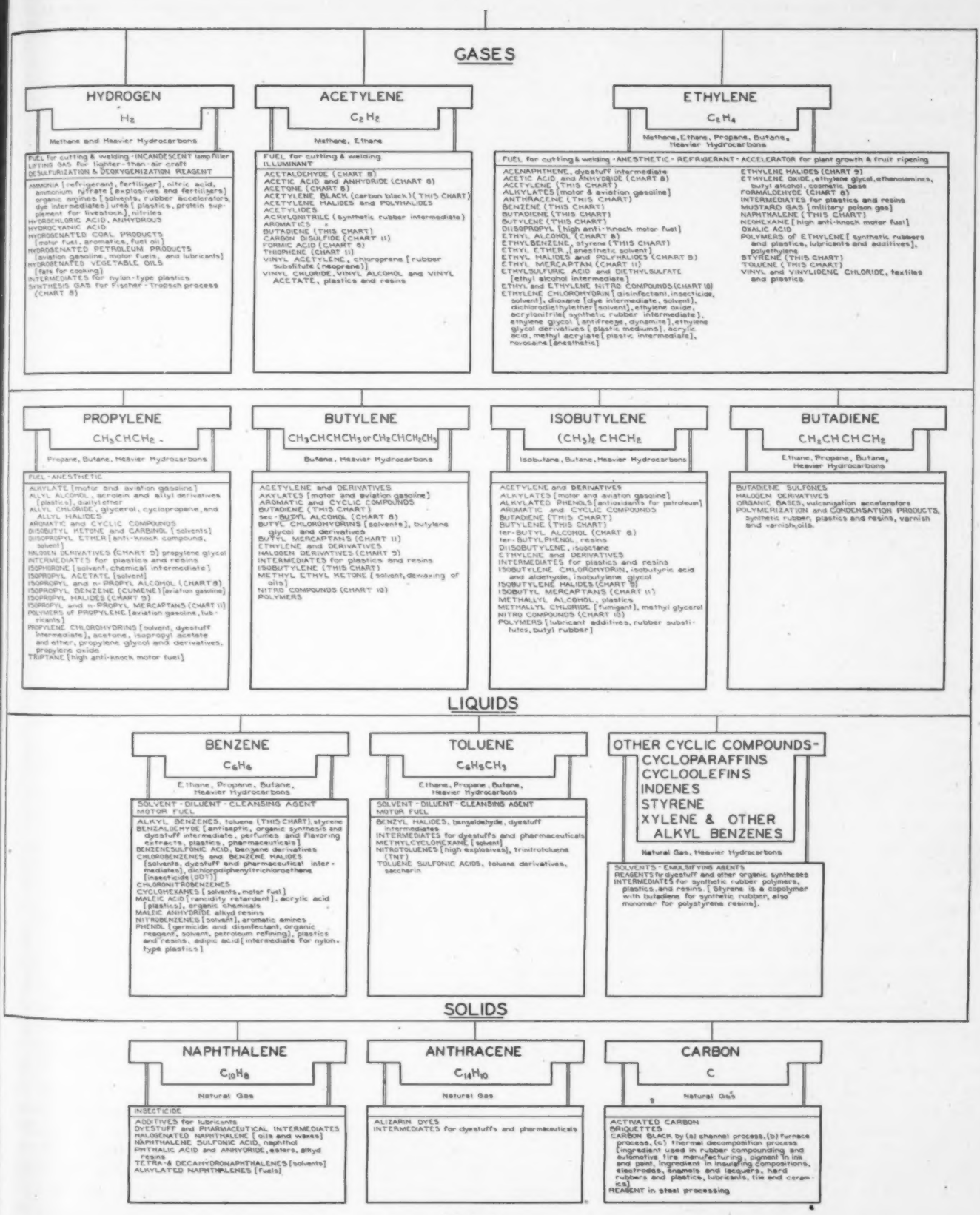
GASES
HYDROGEN
H_2
Methane and Heavier Hydrocarbons
FUEL for cutting & welding · INCANDESCENT lamp filler
LIFTING GAS for lighter-than-air craft
DESULFURIZATION & DEOXYGENIZATION REAGENT
AMMONIA [refrigerant, fertilizer], nitric acid, ammonium nitrate [explosives and fertilizers], organic amines [solvents, rubber accelerators, dye intermediates], urea [plastics, protein supplement for livestock], nitriles
HYDROCHLORIC ACID, ANHYDROUS
HYDROCYANIC ACID
HYDROGENATED COAL PRODUCTS [motor fuel, aromatics, fuel oil]
HYDROGENATED PETROLEUM PRODUCTS [aviation gasoline, motor fuels, and lubricants]
HYDROGENATED VEGETABLE OILS [fats for cooking]
INTERMEDIATES for nylon-type plastics
SYNTHESIS GAS for Fischer-Tropsch process (CHART 8)
ACETYLENE
C_2H_2
Methane, Ethane
FUEL for cutting & welding
ILLUMINANT
ACETALDEHYDE (CHART 8)
ACETIC ACID and ANHYDRIDE (CHART 8)
ACETONE (CHART 8)
ACETYLENE BLACK (carbon black) (THIS CHART)
ACETYLENE HALIDES and POLYHALIDES
ACETYLIDES
ACRYLONITRILE (synthetic rubber intermediate)
AROMATICS
BUTADIENE (THIS CHART)
CARBON DISULFIDE (CHART 11)
FORMIC ACID (CHART 8)
THIOPHENE (CHART 11)
VINYL ACETYLENE, chloroprene [rubber substitute (neoprene)]
VINYL CHLORIDE, VINYL ALCOHOL and VINYL ACETATE, plastics and resins
ETHYLENE
C_2H_4
Methane, Ethane, Propane, Butane, Heavier Hydrocarbons
FUEL for cutting & welding · ANESTHETIC · REFRIGERANT · ACCELERATOR for plant growth & fruit ripening
ACENAPHTHENE, dyestuff intermediate
ACETIC ACID and ANHYDRIDE (CHART 8)
ACETYLENE (THIS CHART)
ALKYLATES [motor & aviation gasoline]
ANTHRACENE (THIS CHART)
BENZENE (THIS CHART)
BUTADIENE (THIS CHART)
BUTYLENE (THIS CHART)
DIISOPROPYL [high anti-knock motor fuel]
ETHYL ALCOHOL (CHART 8)
ETHYLBENZENE, styrene (THIS CHART)
ETHYL ETHER, [anesthetic solvent]
ETHYL HALIDES and POLYHALIDES (CHART 9)
ETHYL MERCAPTAN (CHART 11)
ETHYLSULFURIC ACID and DIETHYLSULFATE [ethyl alcohol intermediate]
ETHYL and ETHYLENE NITRO COMPOUNDS (CHART 10)
ETHYLENE CHLOROHYDRIN [disinfectant, insecticide, solvent], dioxane [dye intermediate, solvent], dichloradiethylether [solvent], ethylene oxide, acrylonitrile [synthetic rubber intermediate], ethylene glycol [antifreeze, dynamite], ethylene glycol derivatives [plastic mediums], acrylic acid, methyl acrylate [plastic intermediate], novocaine [anesthetic]
ETHYLENE HALIDES (CHART 9)
ETHYLENE OXIDE, ethylene glycol, ethanolamines, butyl alcohol, cosmetic base
FORMALDEHYDE (CHART 8)
INTERMEDIATES for plastics and resins
MUSTARD GAS [military poison gas]
NAPHTHALENE (THIS CHART)
NEOHEXANE [high anti-knock motor fuel]
OXALIC ACID
POLYMERS of ETHYLENE [synthetic rubbers and plastics, lubricants and additives], polyethylene
STYRENE (THIS CHART)
TOLUENE (THIS CHART)
VINYL and VINYLIDENE CHLORIDE, textiles and plastics
PROPYLENE
CH_3CHCH_2
Propane, Butane, Heavier Hydrocarbons
FUEL · ANESTHETIC
ALKYLATE [motor and aviation gasoline]
ALLYL ALCOHOL, acrolein and allyl derivatives [plastics], diallylether
ALLYL CHLORIDE, glycerol, cyclopropane, and ALLYL HALIDES
AROMATIC and CYCLIC COMPOUNDS
DIISOBUTYL KETONE and CARBINOL [solvents]
DIISOPROPYL ETHER [anti-knock compound, solvent]
HALOGEN DERIVATIVES (CHART 9) propylene glycol INTERMEDIATES for plastics and resins
ISOPHORONE [solvent, chemical intermediate]
ISOPROPYL ACETATE [solvent]
ISOPROPYL and n-PROPYL ALCOHOL (CHART 8)
ISOPROPYL BENZENE (CUMENE) [aviation gasoline]
ISOPROPYL HALIDES (CHART 9)
ISOPROPYL and n-PROPYL MERCAPTANS (CHART 11)
POLYMERS of PROPYLENE [aviation gasoline, lubricants]
PROPYLENE CHLOROHYDRINS [solvent, dyestuff intermediate], acetone, isopropyl acetate and ether, propylene glycol and derivatives, propylene oxide
TRIPTANE [high anti-knock motor fuel]
BUTYLENE
$CH_3CHCHCH_3$ or $CH_2CHCH_2CH_3$
Butane, Heavier Hydrocarbons
ACETYLENE and DERIVATIVES
ALKYLATES [motor and aviation gasoline]
AROMATIC and CYCLIC COMPOUNDS
BUTADIENE (THIS CHART)
sec-BUTYL ALCOHOL (CHART 8)
BUTYL CHLOROHYDRINS [solvents], butylene glycol and derivatives
BUTYL MERCAPTANS (CHART 11)
ETHYLENE and DERIVATIVES
HALOGEN DERIVATIVES (CHART 9)
INTERMEDIATES for plastics and resins
ISOBUTYLENE (THIS CHART)
METHYL ETHYL KETONE (solvent, dewaxing of oils)
NITRO COMPOUNDS (CHART 10)
POLYMERS
ISOBUTYLENE
$(CH_3)_2CHCH_2$
Isobutane, Butane, Heavier Hydrocarbons
ACETYLENE and DERIVATIVES
ALKYLATES [motor and aviation gasoline]
ALKYLATED PHENOLS [antioxidants for petroleum]
AROMATIC and CYCLIC COMPOUNDS
BUTADIENE (THIS CHART)
BUTYLENE (THIS CHART)
ter-BUTYL ALCOHOL (CHART 8)
ter-BUTYLPHENOL, resins
DIISOBUTYLENE, isooctane
ETHYLENE and DERIVATIVES
INTERMEDIATES for plastics and resins
ISOBUTYLENE CHLOROHYDRIN, isobutyric acid and aldehyde, isobutylene glycol
ISOBUTYLENE HALIDES (CHART 9)
ISOBUTYL MERCAPTANS (CHART 11)
METHALLYL ALCOHOL, plastics
METHALLYL CHLORIDE [fumigant], methyl glycerol
NITRO COMPOUNDS (CHART 10)
POLYMERS [lubricant additives, rubber substitutes, butyl rubber]
BUTADIENE
$CH_2CHCHCH_2$
Ethane, Propane, Butane, Heavier Hydrocarbons
BUTADIENE SULFONES
HALOGEN DERIVATIVES
ORGANIC BASES, vulcanization accelerators
POLYMERIZATION and CONDENSATION PRODUCTS, synthetic rubber, plastics and resins, varnish and varnish oils
LIQUIDS
BENZENE
C_6H_6
Ethane, Propane, Butane, Heavier Hydrocarbons
SOLVENT · DILUENT · CLEANSING AGENT
MOTOR FUEL
ALKYL BENZENES, toluene (THIS CHART), styrene
BENZALDEHYDE [antiseptic, organic synthesis and dyestuff intermediate, perfumes and flavoring extracts, plastics, pharmaceuticals]
BENZENESULFONIC ACID, benzene derivatives
CHLOROBENZENES and BENZENE HALIDES [solvents, dyestuff and pharmaceutical intermediates], dichlorodiphenyltrichloroethane [insecticide (DDT)]
CHLORONITROBENZENES
CYCLOHEXANES [solvents, motor fuel]
MALEIC ACID [rancidity retardant], acrylic acid [plastics], organic chemicals
MALEIC ANHYDRIDE alkyd resins
NITROBENZENES [solvent], aromatic amines
PHENOL [germicide and disinfectant, organic reagent, solvent, petroleum refining], plastics and resins, adipic acid [intermediate for nylon-type plastics]
TOLUENE
$C_6H_5CH_3$
Ethane, Propane, Butane, Heavier Hydrocarbons
SOLVENT · DILUENT · CLEANSING AGENT
MOTOR FUEL
BENZYL HALIDES, benzaldehyde, dyestuff intermediates
INTERMEDIATES for dyestuffs and pharmaceuticals
METHYLCYCLOHEXANE [solvent]
NITROTOLUENES [high explosives], trinitrotoluene (TNT)
TOLUENE SULFONIC ACIDS, toluene derivatives, saccharin
OTHER CYCLIC COMPOUNDS-
CYCLOPARAFFINS
CYCLOOLEFINS
INDENES
STYRENE
XYLENE & OTHER ALKYL BENZENES
Natural Gas, Heavier Hydrocarbons
SOLVENTS · EMULSIFYING AGENTS
REAGENTS for dyestuff and other organic syntheses
INTERMEDIATES for synthetic rubber polymers, plastics and resins. [Styrene is a copolymer with butadiene for synthetic rubber, also monomer for polystyrene resins].
SOLIDS
NAPHTHALENE
$C_{10}H_8$
Natural Gas
INSECTICIDE
ADDITIVES for lubricants
DYESTUFF and PHARMACEUTICAL INTERMEDIATES
HALOGENATED NAPHTHALENE [oils and waxes]
NAPHTHALENE SULFONIC ACID, naphthol
PHTHALIC ACID and ANHYDRIDE, esters, alkyd resins
TETRA- & DECAHYDRONAPHTHALENES [solvents]
ALKYLATED NAPHTHALENES [fuels]
ANTHRACENE
$C_{14}H_{10}$
Natural Gas
ALIZARIN DYES
INTERMEDIATES for dyestuffs and pharmaceuticals
CARBON
C
Natural Gas
ACTIVATED CARBON
BRIQUETTES
CARBON BLACK by (a) channel process, (b) furnace process, (c) thermal decomposition process [ingredient used in rubber compounding and automotive tire manufacturing, pigment in ink and paint, ingredient in insulating compositions, electrodes, enamels and lacquers, hard rubbers and plastics, lubricants, tile and ceramics]
REAGENT in steel processing

CHART 7

Products obtainable from natural gas by decomposition (thermal-catalytic-electric).

carbon materials, but has been obtained from natural-gas hydrocarbons by thermal decomposition or by use of electric-arc cracking.

Ethylene is an important constituent in refinery gas produced in the manufacture of gasoline from nongaseous petroleum hydrocarbons. It is readily produced by pyrolysis of natural gas. Propylene, butylene, and isobutylene are produced similarly. These unsaturated hydrocarbons are of the type known as olefins and form the basis for the manufacture of a broad series of organic chemicals. Ethylene, which has been termed a "building block" for organic synthesis, is the starting point for the commercial production of more than 50 products. A correspondingly large number of products is obtained from propylene, butylene, and isobutylene. In the manufacture of aviation fuels and alkylates, these olefins are of prime importance. The production of butadiene has been discussed in Chart 5.

Besides heavier aliphatic hydrocarbons, not included separately in the chart, that may be formed as the result of decomposition of natural-gas hydrocarbons, a group of cyclic compounds may be formed, which include benzene, toluene, xylene, and other aromatic compounds. However, selected cuts of heavier hydrocarbons from petroleum used as feed stock in catalytic aromatization gives high yields of benzene or toluene, and much of the toluene needed for wartime demands was obtained by this method.

The use of natural gas for the production of carbon black was one of its first non-energy uses. Three methods, known as the channel, thermal decomposition, and furnace processes, are used for the production of carbon black. Each process yields a product of different type especially suited for specific uses. Carbon black is an essential ingredient in the manufacture of automobile tires and other rubber goods and in the production of paints, inks, and other materials. A type of carbon black known as "acetylene black" is produced by the electrical decomposition of acetylene.

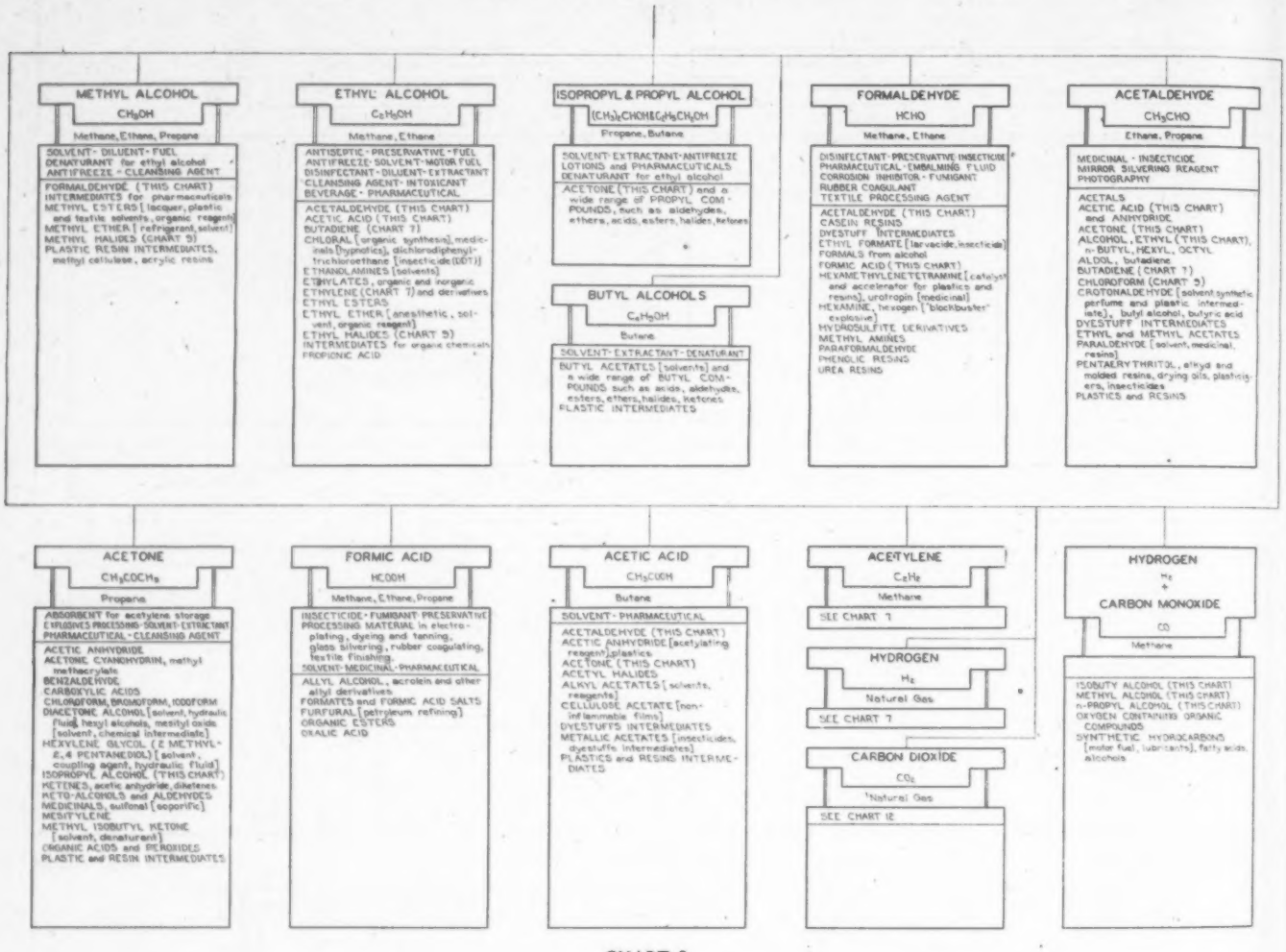

CHART 8

Products obtainable from natural-gas hydrocarbons by oxidation (thermal-catalytic-pressure).

Chart 8

Oxidation of the hydrocarbon in natural gas in the form of combustion furnishes the basis for its use as a heat and energy source. The ultimate products of combustion of hydrocarbons are carbon dioxide and water. Controlled and limited oxidation of hydrocarbons produces a number of products highly useful in the production of textiles and plastics and in other industries. Among the oxidation products obtainable from natural gas and other petroleum hydrocarbons are alcohols, acids, fatty acids, and aldehydes. Those of most importance produced commercially from natural gas include methyl, ethyl, isopropyl, propyl, and butyl alcohols, formaldehyde and acetaldehyde, acetone methylacetone, acetic acid, acetic anhydride, propionic acid, and many derivatives. Both saturated and unsaturated aliphatic hydrocarbons are utilized, ordinarily in the presence of a catalyst, in the production of oxidation products.

The reaction of natural-gas hydrocarbons with

Natural gas hydrocarbons can undergo direct fluorination, chlorination, bromination, and iodination when reacted under proper conditions with halogens or halogen-containing compounds. Direct fluorination is said to be violent and difficult to control but is reported accomplished with methane, ethane, and other hydrocarbons. Iodination is a reversible reaction. Bromination and chlorination are effected most easily. Olefin hydrocarbons generally react easily with halogens, forming addition compounds; substitutive halogenation takes place under certain conditions, forming allylic halides (Chart 7).

Halogen atoms in aliphatic halides may be replaced by halogens preceding in the atomic series. Fluoro- or fluorochlorohydrocarbons usually are obtained by replacement of chlorine with fluorine in chlorohydrocarbons.

Products of chlorination only are shown in this chart as representative of products obtained by halogenation of natural gas hydrocarbons, as the greatest laboratory and commercial attention has been given to chlorination.

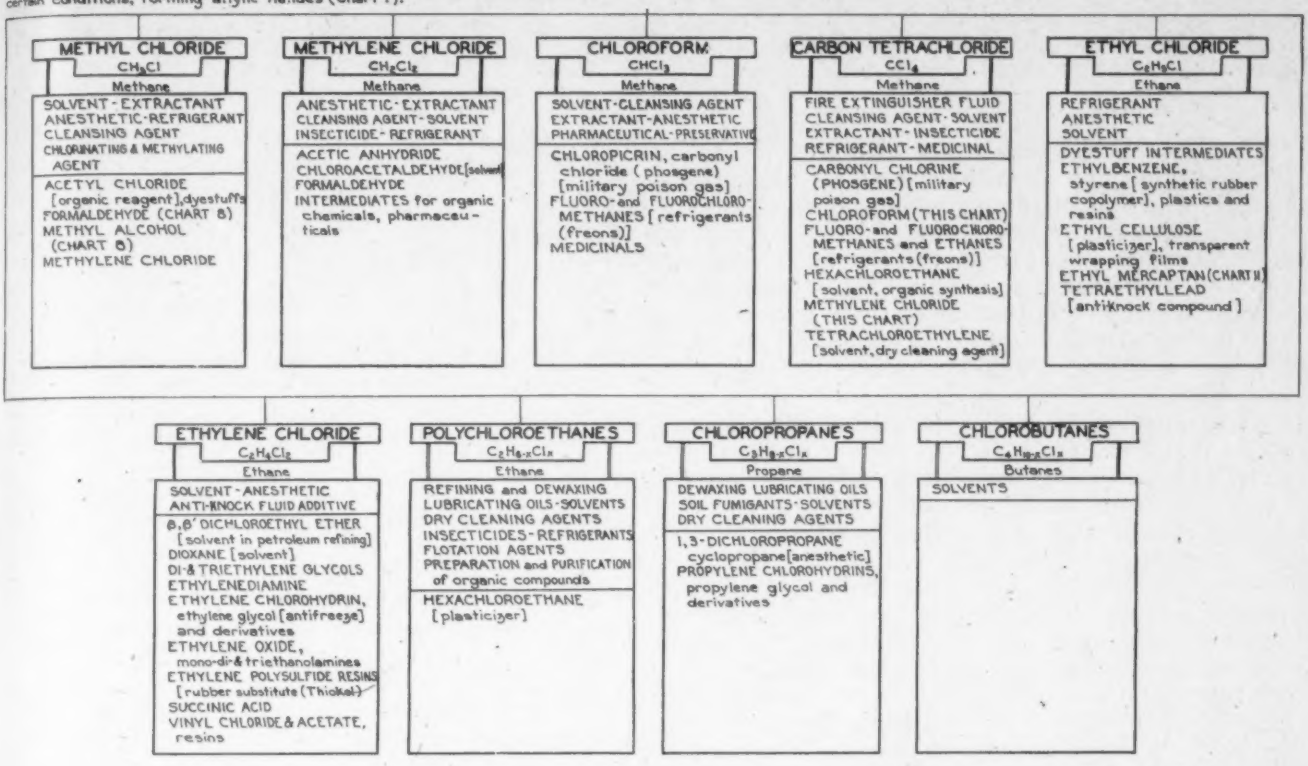

CHART 9

Products obtainable from natural-gas hydrocarbons by halogenation (thermal-catalytic-photolytic).

steam, oxygen, and carbon dioxide to form a "synthesis gas" for the Fischer-Tropsch synthesis of higher hydrocarbons has received much recent attention as a possible method for utilization of methane in the manufacture of motor fuels. In addition, organic chemicals such as alcohols, aldehydes, acids, and esters can be produced concurrently with the motor fuel.

The reaction of methane and steam when carried to completion gives carbon dioxide and hydrogen. This reaction has been the basis for production of hydrogen for many of the plants that produced ammonia and nitric acid for wartime use. Natural gas or hydrogen from natural gas also is used in the manufacture of pure nitrogen from air for use in ammonia manufacture. By means of controlled combustion, the oxygen content of air is removed, leaving nitrogen, carbon dioxide, and water, which are separated easily. Possible by-products and derivatives besides ammonium nitrate in the manufacture of ammonia and nitric acid include methyl alcohol from hydrogen and carbon monoxide, formaldehyde from methyl alcohol, dry ice from waste carbon dioxide, urea from liquid ammonia and carbon dioxide, and perhaps ammonium sulfate, sodium carbonate, sodium nitrate, dicalcium phosphate, hydrogen cyanide, formamide, and others.

Chart 9

Chart 9 shows products of chlorination as representatives of products obtainable by halogenation of natural gas. Chlorinated hydrocarbons form an important class of chemicals, which are used directly as solvents, refrigerants, degreasing compounds, and for many other purposes; also as intermediates in organic chemical synthesis. Fluoro and fluorochloro compounds, which are used principally as refrigerants, usually are obtained by replacement of chlorine with fluorine in a chloro compound. A few brominated hydrocarbons are in commercial production. Methyl bromide is used as a fumigant and insecticide. Ethylene dibromide is used in tetraethyl lead fluids, as a solvent, and as an intermediate in the production of a number of chemicals.

Chart 10

Active research into the nitration of natural-gas hydrocarbons began around 1930 and has produced

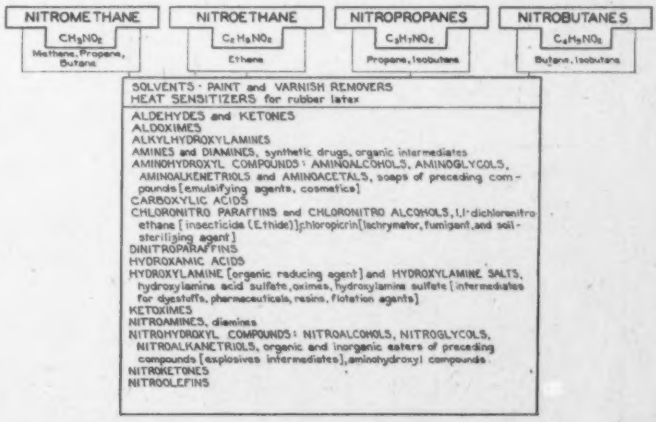

CHART 10

Products obtainable from natural-gas hydrocarbons by nitration (thermal).

a variety of products. Commercial production of nitroparaffins by use of the vapor-phase nitration reaction of nitric acid and hydrocarbons from petroleum and natural gas, occurred within the present decade. Uses for these nitroparaffins and their derivatives in many fields already have been developed, and other extensive uses have been prophesied.

Chart 11

Chart 11 shows products obtainable from natural gas and other petroleum hydrocarbons by sulfurization. Production of these products is small, but recent announcements indicate future commercial-scale manufacture of carbon disulfide, thiophene, mercaptans, thioethers, and other sulfur compounds, using natural gas and sulfur or hydrogen sulfide as starting materials. Butyl mercaptan and butyl disulfide are said to be available already in tank-car lots, and other sulfur compounds are being produced in experimental or pilot-plant quantities. One company states that a plant was erected and operated successfully for the manufacture of tertiary dodecyl mercaptan (C_{12}), using as raw materials hydrogen sulfide derived from natural gas, with copolymers from propylene and isobutylene resulting from thermal decomposition of isobutane. The plant was built in anticipation of a demand for the product in the synthetic-rubber program but was shut down when the demand did not materialize. Another company has announced the pilot-plant and contemplated commercial production of thiophene. Still another company has announced the pilot-plant development of a process for the manufacture of carbon disulfide by the catalytic reaction of sulfur and hydrocarbons from natural gas.

Many of the uses for sulfurization products shown in Chart 11 are those suggested by manufacturers as possible uses and have not been proven commercially.

Chart 12

Non-hydrocarbon constituents of natural gas usually are present in relatively small amounts. Some of these materials, such as helium, are of enough value to warrant their recovery even when present in quantities of only a few percent. Others, such as hydrogen sulfide, usually are objectionable and ordinarily must be removed before the hydrocarbon content of the gas can be utilized. Hydrogen sulfide and other objectionable gases may become valuable when separated and used in the production of other products. The use of hydrogen sulfide in the production of sulfur compounds from hydrocarbons already has been mentioned. In at least one natural-gas field it is recovered and used for the production of elemental sulfur.

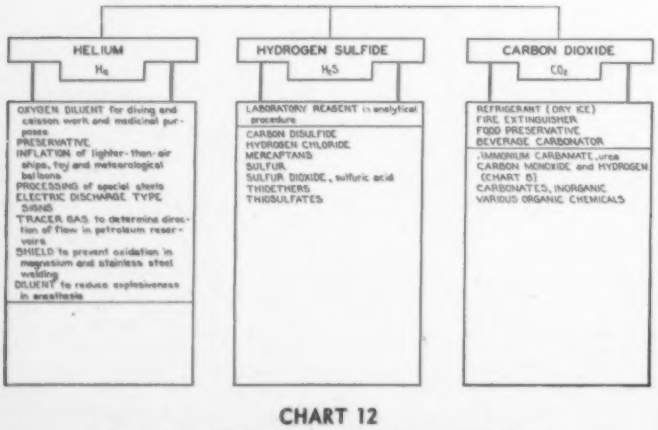

CHART 12
Uses of non-hydrocarbon natural-gas constituents.

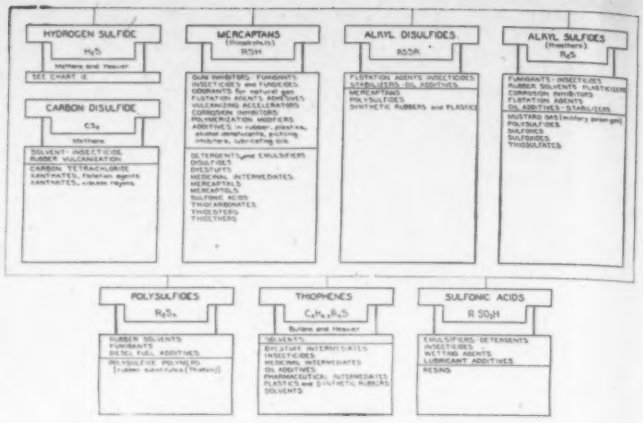

CHART 11
Products obtainable from natural gas and other petroleum hydrocarbons by sulfurization.

Some natural gases are composed almost entirely of carbon dioxide. Carbon dioxide from this source may be of value if the wells from which it is produced are geographically situated where the gas may be utilized economically. So far, no economic use for nitrogen in natural gas has been developed.

Acknowledgments

Before starting revision of this paper, a number of commercial organizations known or believed to be interested in the field of natural gas utilization were invited to contribute data and suggestions. The replies received were materially helpful in the compilation and preparation of the report, and the writers are pleased to acknowledge their indebtedness to the members of these organizations for their contributions.

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Instrumentation in Gas-Cycling Plants

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SELECTION of the proper instrumentation for a given process application is governed by the same basic concepts for essentially all continuous processes. There are, however, special requirements, or limitations, imposed by the various types of operation to which a given process step may be adapted, which may influence the selection of controls to such an extent that individually different control arrangements may be required for a given application, depending on whether in an oil-refining, petro-chemical or gas-cycling operation. It is intended to limit the scope of this article to consideration of some of the special factors influencing the design of control systems for gas-cycling plants.

Processes common to gas-cycling operations generally are not complex and are characterized by steady operating conditions; thus their instrumentation usually is relatively simple and straightforward. It must be recognized, however, in selecting controls for such a plant that personnel and facilities available for the supervision and maintenance of control equipment probably will not be of the caliber customarily found in oil refineries and chemical plants. Simplification is desirable to reduce instrument supervision and maintenance requirements to the bare minimum, while, on the other hand, relatively complete instrumentation is desirable to maintain positive control. Probably the better compromise is in favor of complete instrumentation if every reasonable precaution is taken in selecting equipment and

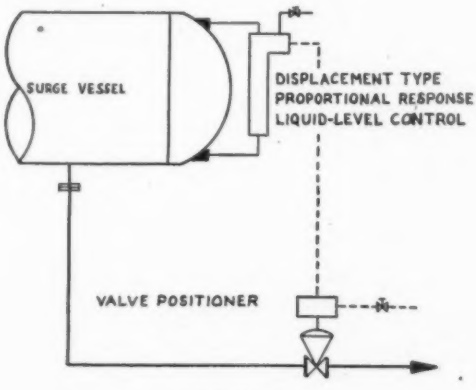


FIGURE 1

making proper installations to minimize the supervision required.

Although the importance of proper field installation is admittedly great, this phase of the problem has been thoroughly explored by others, and lies beyond the scope of this discussion. Let it suffice to say here that the construction forces have a definite contribution to make in reducing the instrument-maintenance problems of the operating group.

Design of control systems for a new facility usually develops along with, or follows immediately, the completion of the process design. This involves analyzing the various applications to determine the type of control most desirable. The analysis of applications is followed by selection of actual equipment to do the job. It is at this stage that the control engineer can make a real contribution toward reducing the instrument maintenance and supervision worries of the plant operator.

Of primary importance is the provision of a good supply of operating medium for the pneumatic controls. Natural gas commonly is used for this purpose in cycling plants, although compressed air is sometimes provided; either should be thoroughly filtered and dried, preferably, before distribution to the various instruments, and should be supplied at constant pressure. Further, where natural gas is used, careful checks for the presence of hydrogen sulfide should be made, even though the main gas stream to the plant is considered sweet. If hydrogen sulfide is present, suitable treatment for its removal should be given the gas before entering the dryers. Further

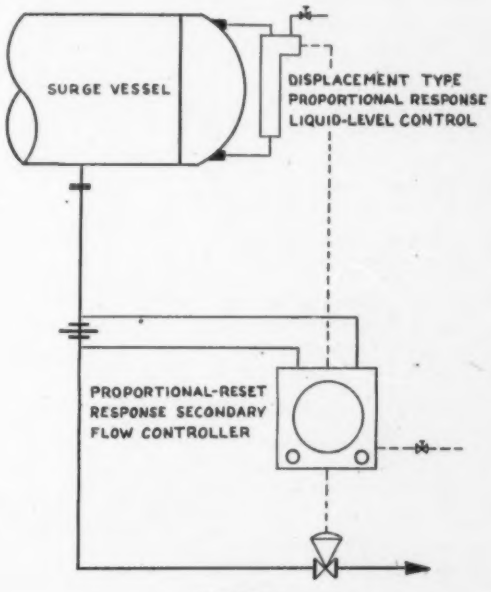


FIGURE 2

responsibility of the control engineer is to make certain that the process design fulfills such needs as:

(a) Surge capacities where necessary, and in at least the volumes required, for control purposes;
(b) Line sizes amply large to accommodate control valves and meter runs;
(c) Pressure differentials adequate to assure the effectiveness of control valves, etc.

In the selection of control equipment, maintenance

and supervision requirements can be reduced in some instances by minor sacrifices in the possible approach to ideal precision of control.

Consider, as an example, the problem of controlling the liquid level in a rich-oil flash tank by regulating

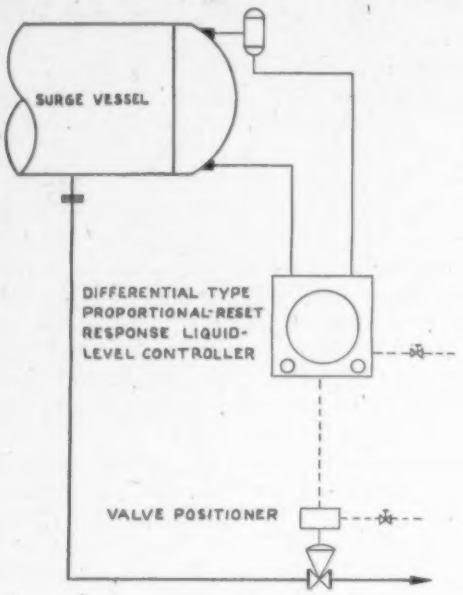


FIGURE 3a

the outflow of oil. Assume that "averaging" control definitely is desired; that is, the controller should make definite use of surge time available in the vessel to absorb variations in rate of flow of incoming oil in order to maintain, as nearly as possible, a uniform rate of flow of oil from the vessel. An installation such as is shown in Figure 1 would fulfill this requirement. If a flow record is wanted, or if the pressure differential across the control valve

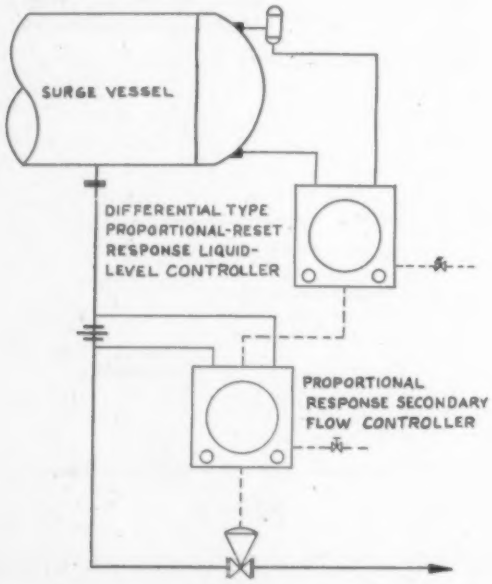


FIGURE 4a

is variable, the arrangement in Figure 2 may be more desirable. Frequently, it also is required to "average" the level essentially about a fixed control point, say the centerline of the vessel. This usually is desirable in absorption-type cycling plants, since the absorption oil circulates in a closed cycle and persistent level variations in any given surge vessel must be compensated by level changes at other surge points in the system. To effect this type of control it is necessary to incorporate reset response in the control system. Usually this control feature is added in the mechanism of the primary measuring device, in this case the liquid-level controller. Typical arrangements are given in Figures 3a and 3b, which show, respectively, differential- and displacement-type proportional reset response level controllers actuating control valves. Since controllers of this type frequently operate with a relatively wide proportional band

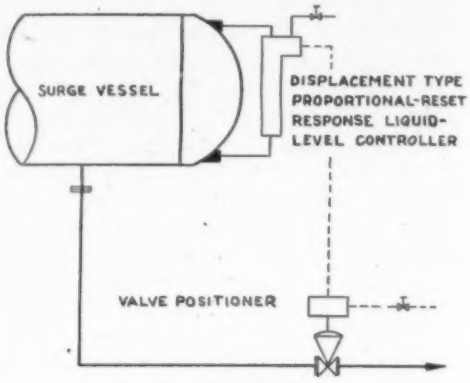


FIGURE 3b

adjustment (as high as 150 percent, or more, of full scale for instruments provided with auxiliary limit control and 80-100 percent for those without) it is essential that the controlled valve follow the controller almost perfectly; thus, the valve positioners indicated on the drawings are almost a necessity. In applications where a flow record is required or variable pressure drop across the control valve dictates the use of a secondary flow controller, as illustrated in Figures 4a and 4b, the positioner is no longer a basic requirement. It may, of course, be desirable to

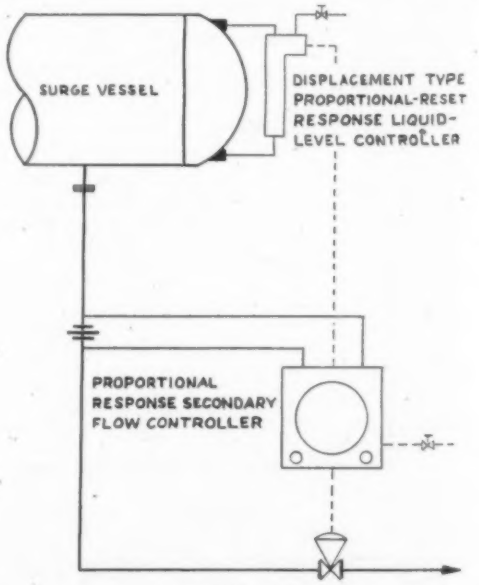


FIGURE 4b

provide a positioner to assist the flow controller for the usual reasons; for example, if the control valve is relatively large, or operates under conditions which result in heavy stem or guide friction, or extreme unbalance of the plug.

Although both the differential and displacement types have their ardent proponents, each type pro-

vides the same basic elements of level control. The differential type probably affords the closer approach to an ideal "averaging" instrument, whereas the displacement type is probably somewhat easier to maintain and is almost fool-proof by comparison. Because of the simpler maintenance requirement, the displacement type usually will be the preferable selection for cycling applications. This will be particularly true for services in liquids having a bubble-point near the ambient temperature at operating pressure; e.g., rich-oil and distillate flash tanks and reflux accumulators.

Figure 5 is directed to those proponents of displacement type controls whose favorite make of instrument is not available with proportional-reset

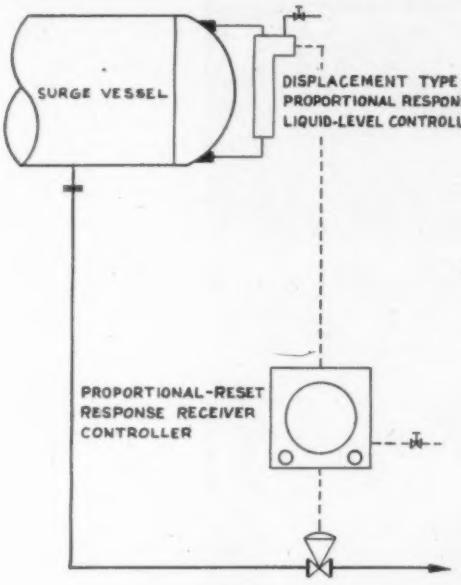


FIGURE 5

response. Shown is a simple proportional response level controller used as pneumatic transmitter for a proportional-reset response receiver-controller, which in turn actuates the control valve.

In designing flow-control systems to operate with the minimum maintenance requirement perhaps the most important single factor is to arrange for the meter body to be installed relatively near the orifice. This applies whether the meter body is on a flow transmitter or a direct-connected controller, and means that the length of any direct connected system should be limited to the absolute minimum.

Temperature-control systems employing fluid-filled capillary systems for measurement in the primary device should be similarly treated. Minimum length of capillary tubing will assure maximum responsiveness for a given system and will reduce vulnerability to physical damage. Owing to the relatively great measuring lag inherent in most temperature measurements, temperature controllers normally operate with rather broad proportional band settings and, as a result, their controlled valves usually require a positioner, pressure balancing or a secondary flow controller to minimize control lag.

Temperature Measurement

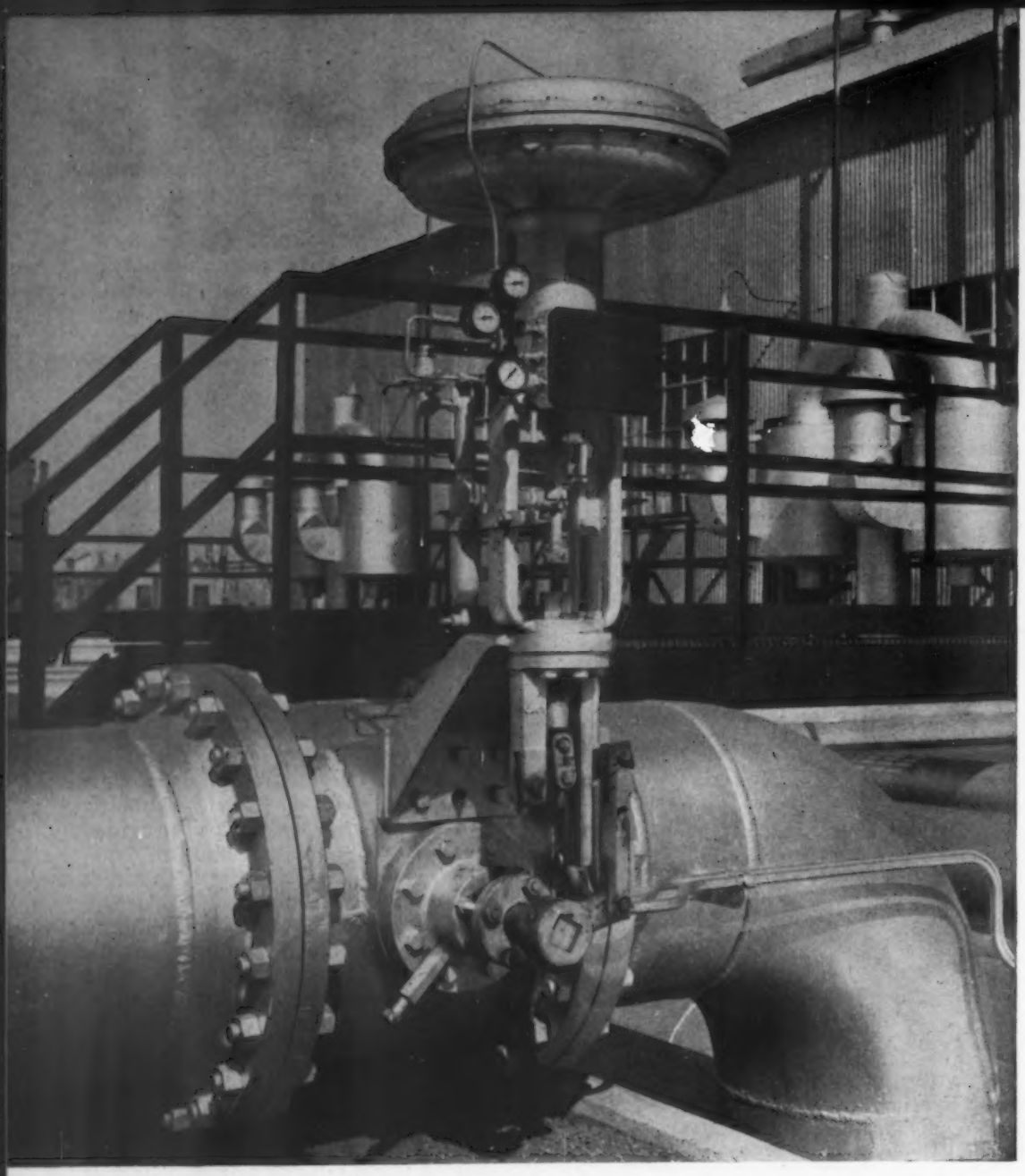
Although electrical measurement of temperature generally is considered to be the most desirable method to use in the control of fractionators making precision separations, such as deisobutanizers and deisopentanizers for example, many such columns were installed during recent years in cycling plants using fluid filled capillary systems for temperature measurement. In most cases this probably was done to assure continuity of operation, since the prime movers throughout most such plants are steam-driven and the source of electrical power usually is from a small auxiliary power plant not sufficiently reliable for process controls to be dependent upon it.

Pressure-control applications in cycling installations usually permit the use of direct-connected systems without complications.

Since it is not practical to arrange for all process orifice-meter runs or temperature-bulb locations to be near a central control panel, it follows that the primary devices, if direct connected, should be located near the equipment they serve. Alternatively, if it is desired to assemble all instruments on a centrally-located control panel, the measurement should be transmitted from a point near the orifice or temperature bulb to a receiver-controller on the control panel. Transmission of a measurement from the primary device to the receiver-controller may be accomplished either pneumatically or electrically; however, in plants of the type under consideration, pneumatic transmission is used almost without exception. Many advocates of the dispersed control arrangement referred to above base their preference, at least partially, upon prior unhappy experience with pneumatic transmitters. Actually, plants which are provided with a properly controlled source of operating medium, as postulated above, suffer very little from mechanical difficulties in transmitters. Frequently cited also is the difficulty experienced in maintaining absolute calibration between the transmitter and the receiver-controller. This is largely a self-inflicted difficulty, because in cycling operations there is no real need for knowing, with absolute accuracy, the magnitude of any given operating variable. Yield or accounting meters, of course, are excluded for this category since they properly should be direct connected units located at their respective orifice-meter runs. Those favoring the dispersal of controls to the immediate location of the equipment served believe that such an arrangement makes it necessary for the operators to make frequent trips through the operating area in order to observe the control instruments. It appears more logical to acknowledge that the operation of such a plant is basically automatic, in most cases, and that the probability that desired manual adjustments will be made as required, will improve with the convenience of the operation. On this basis, the instruments should be conveniently grouped on a common control panel at a point near the "center of effort" in the operating area. Such an arrangement has other advantages:

1. Control of plant during operational upsets is greatly facilitated.
2. Starting-up and shutting-down operations are greatly simplified.
3. Adequate all-weather protection for both operating personnel and control instruments can be provided more conveniently and probably less expensively.

The instrumentation engineer has a final responsibility in following the mechanical design of the plant facilities to make certain that control valve, pilot-operated regulator, secondary controller and yield meter in locations are conveniently accessible for operating and maintenance requirements.



One of the butterfly valves and valve positioner actuated by the pressure recording-controller instrument to maintain constant gas-intake pressure of 12.2 pounds.

Dual Control System

Synchronizes Compressor Speed-Intake Gas Pressure

J. M. SAPPINGTON
Plant Superintendent,
Absorption Plant, Inc.

MAINTAINING a constant intake pressure to the compressors is a major problem in gasoline plants. Constant intake pressure is especially important in fields where the gas flow fluctuates due to flowing schedules and changes in oil allowables imposed by proration, which varies from day to day and from month to month. In many plants, elaborate dispatching systems have been designed and installed, but have not been entirely effective in solving this problem. In fields where low separator operating pressures are imperative to good yields, it is well worth while to consider the installation of adequate control equipment to take care of this situation.

At Absorption Plant, Inc., Winnie, Texas, 30,000,000 cubic feet daily of low-pressure separator gas is gathered from four separate oil fields in addition to 55,000,000 cubic feet daily of high-pressure gas from gas-distillate wells. There is a large number of wells involved, and due to the fact that they are located so far from the plant, it is impossible to literally "ride herd" on the entire 80-mile field gathering system.

To solve the problems of intake pressure and compressor speed, an automatic speed governor and pressure control system for compressor operation was designed and installed. It is important to note that this dual system is regarded as unusual, despite the fact that all the equipment is conventional, because of the method of application and the resulting efficient performance. A number of operators engaged in constructing new plants and having difficulties with

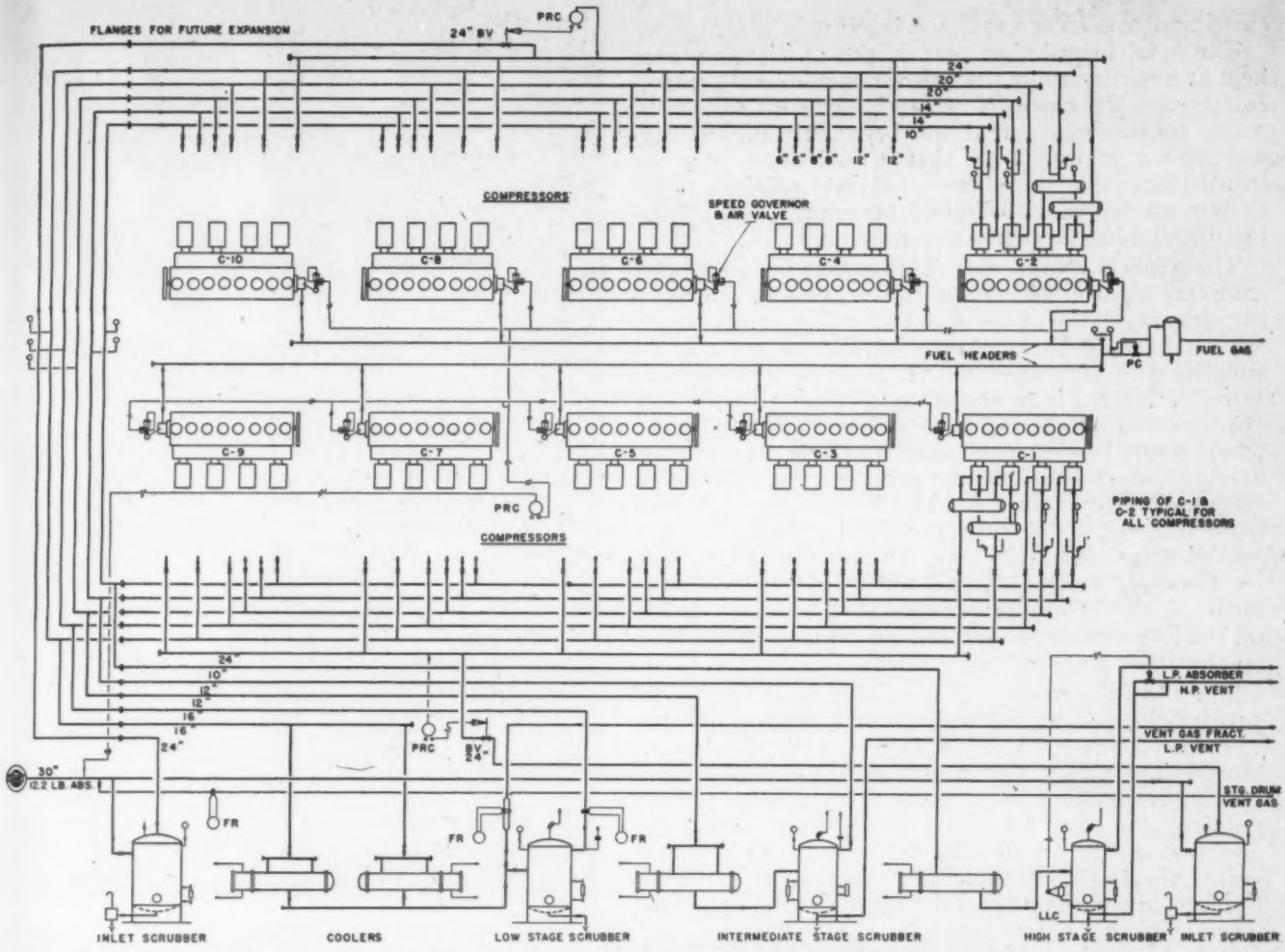
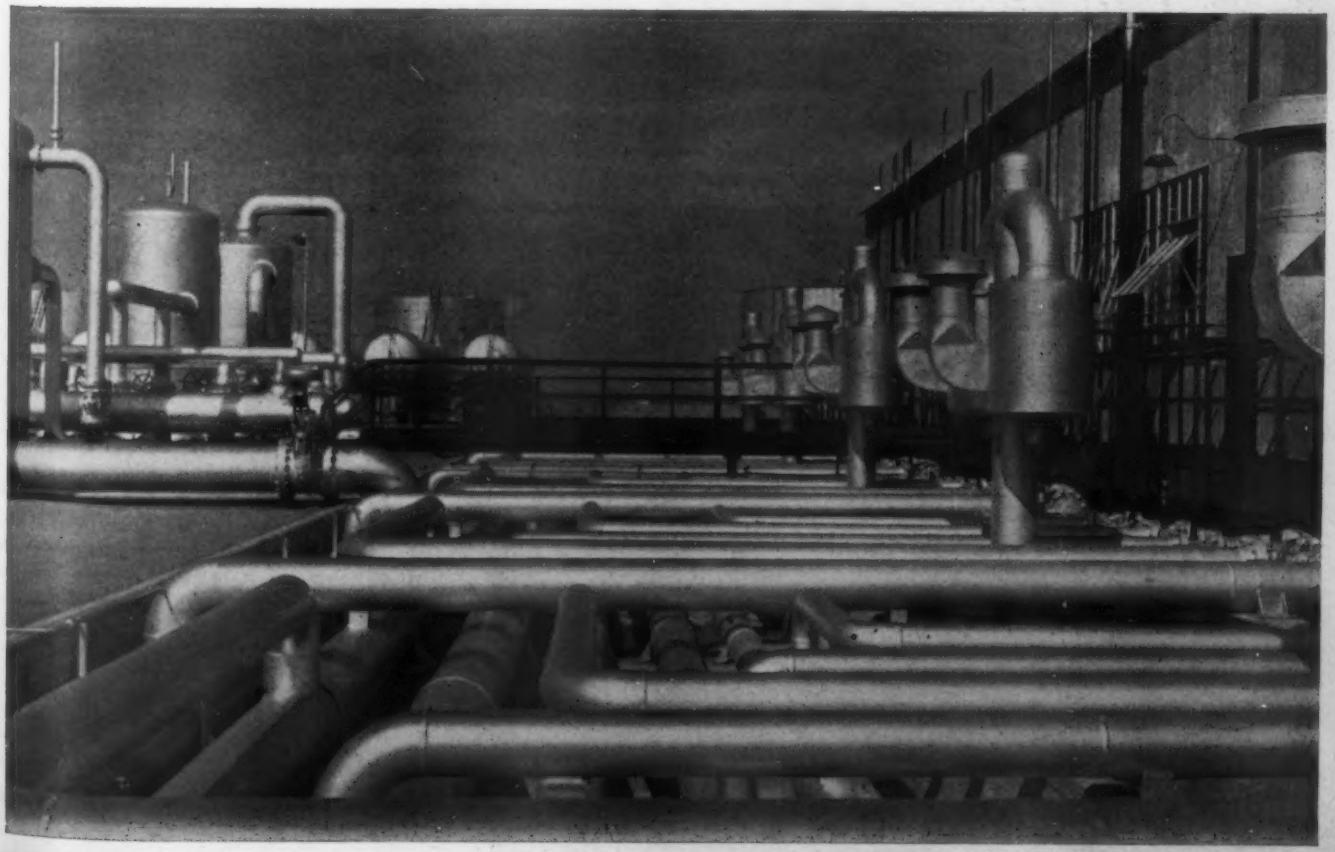


Diagram of compressor installation, showing piping, manifolding, control systems, scrubbers, and shell-and-tube coolers.

Below: Intake and discharge lines for one bank of compressors, manifolded for maximum flexibility and simplicity.

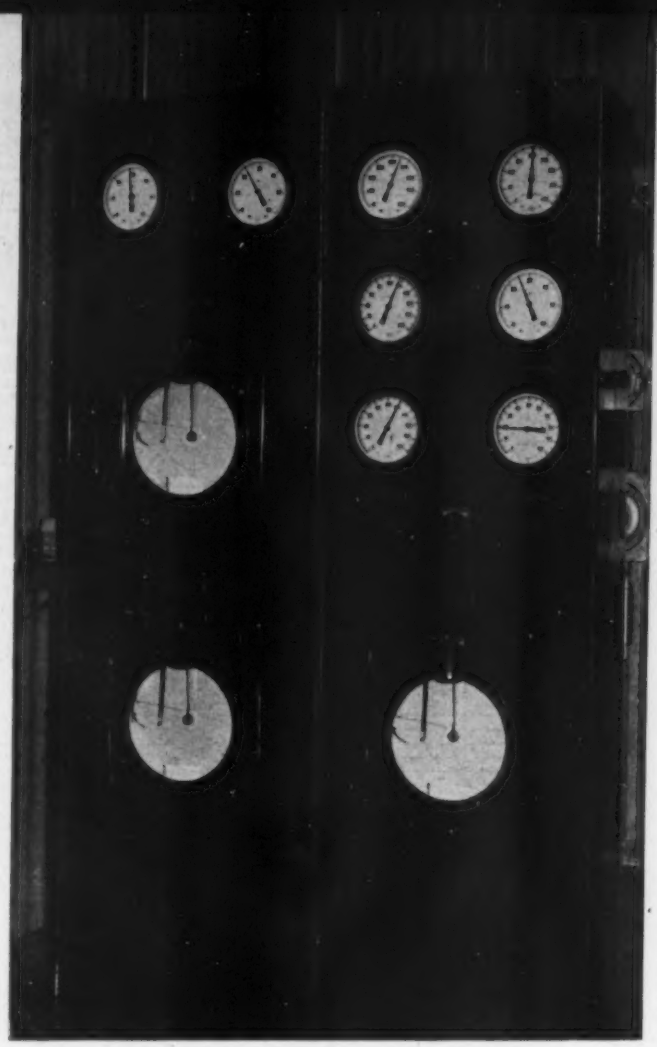


existing plants have expressed interest in the system.

The total installation cost of the application was kept at a minimum by the use of conventional control equipment. This control scheme has paid for itself many times since operations began by eliminating compressor maintenange that would occur due to overloading the machines, enabling efficient field system operation with limited personnel, and reducing dispatching problems to a minimum.

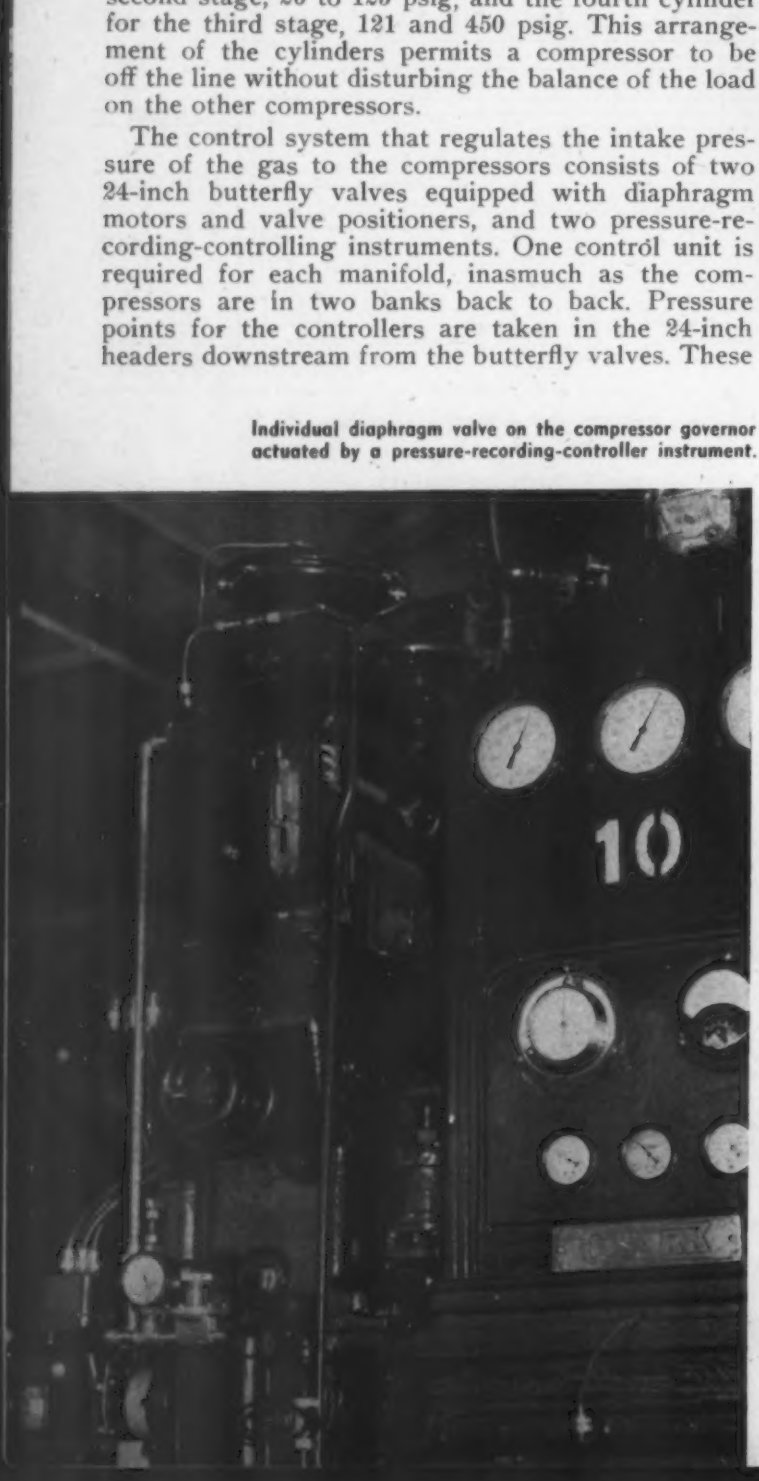
The general compressor plant layout including the complete control hook-up is shown in an accompanying drawing. It will be noted that the compressors are set back to back to save building space and to simplify and keep operation expense at a minimum. For this layout, it is necessary to have two suction headers and intake scrubbers. The ten 800-horsepower angle-type compressors have four compression cylinders each, and compress gas through three stages. Two cylinders for the first stage compress from 12.2 psia to 30 psig, the third cylinder for the second stage, 26 to 125 psig, and the fourth cylinder for the third stage, 121 and 450 psig. This arrangement of the cylinders permits a compressor to be off the line without disturbing the balance of the load on the other compressors.

The control system that regulates the intake pressure of the gas to the compressors consists of two 24-inch butterfly valves equipped with diaphragm motors and valve positioners, and two pressure-recording-controlling instruments. One control unit is required for each manifold, inasmuch as the compressors are in two banks back to back. Pressure points for the controllers are taken in the 24-inch headers downstream from the butterfly valves. These



Main compressor control panel, showing speed governor and butterfly valve recording-controlling pressure instruments.

Individual diaphragm valve on the compressor governor actuated by a pressure-recording-controller instrument.



controllers are set to maintain a gas intake pressure of 12.2 psia to the first stage of compression.

A third pressure-recording-controlling instrument, having a pressure point on the main incoming-gas line ahead of the point where the line divides for the twin inlet scrubbers and manifolds, controls the air pressure to the speed governors and air valves on each compressor. From the diagram, it can be seen that the control lines from the speed governors on each of the five compressors in each bank are connected in a parallel system. Then the controlling air line from the PRC is connected to these two systems. This method assures equal pressure to each speed governor. When the gas load and the intake pressure decrease, the compressors are automatically slowed down. As the gas load and the intake pressure increase, the compressors speed up and maintain 12.2 psia pressure at the intake. The air supply from the PRC to the individual diaphragm valves is set by a valve-positioner instrument so that 13 pounds of air will run the compressors at exactly 325 rpm which is the maximum intended. In case the gas load increases to the extent that all machines running at maximum speed are unable to handle the total volume, the two 24-inch butterfly valves maintain a constant intake pressure, never overloading the compressors.

In addition to the dual-system described above, a third system controls the fuel gas to the compressors. It serves to maintain a constant pressure on the twin fuel manifolds from the fuel header. Incoming fuel gas flows through a drip pot through a pressure regulating valve to the header.

Gas Absorption-Dehydration Plant Processes 150,000,000 Cubic Feet Daily

C. C. PRYOR, Associate Editor

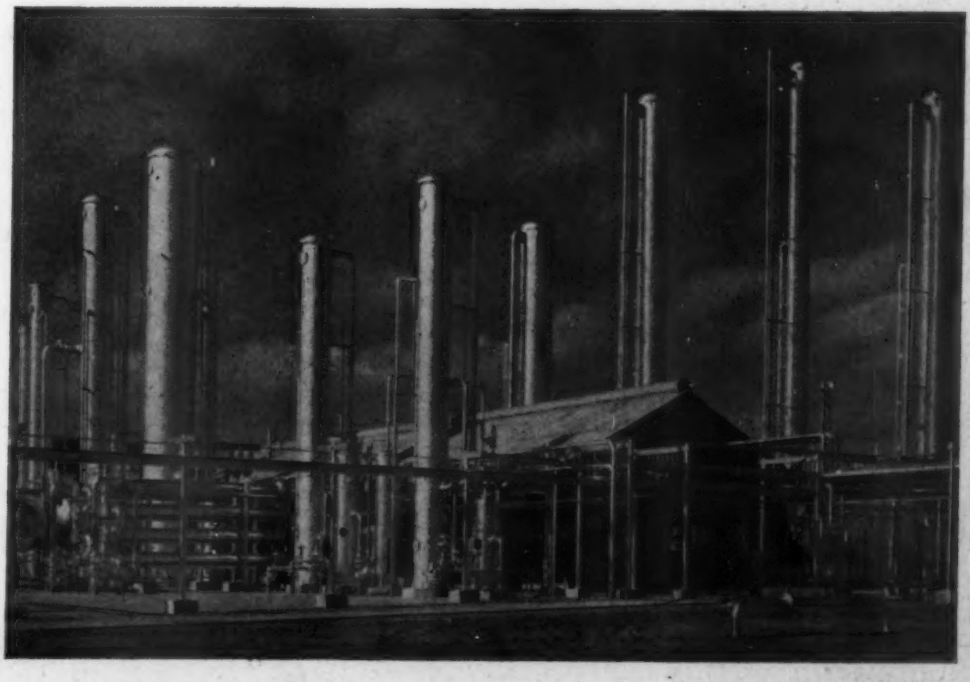
A COMBINATION gasoline-dehydration plant capable of processing and dehydrating 150,000,000 cubic feet daily of natural gas has been placed on stream in the Carthage field of East Texas. This is the Feazel-Kinsey Plant of The Chicago Corporation. The dehydration and absorption equipment are designed for 150,000,000 cubic feet daily of gas and over 3000 barrels of liquid hydrocarbons daily, while the distillation unit is designed to process the hydrocarbons from 125,000,000 cubic feet of gas daily.

Noteworthy features in the design of the plant's process includes high-pressure absorption, high- and low-pressure reabsorption, a de-ethanizer, main and primary stills, gasoline and kerosine fractionators, a condensate and an absorption-oil-purifying still. In addition, three direct-fired tubular heaters are utilized in the process for the rich oil, oil purifying system, and fractionating still.

The dehydration unit of the plant consists of six high-capacity solid-absorbent type, contactor towers, three of which are regenerated under line pressure, while the remaining three are on stream. The unit maintains a dew-point of — 25° F. or lower on the residue gas for pipe-line sale. Gas enters the plant at approximately 1800 psig and is reduced to 1250 psig, which is sufficient pressure for processing and dehydration and to supply sales gas to the pipe lines without compression.

Three steam systems are used in the plant. Steam at 300 psig is used for stripping in the primary and condensate stills and for the turbine-driven centrifugal pumps. This steam is exhausted into the 125 psig system for use in the reboilers and reciprocating pumps. In turn, this steam is exhausted at 15 psig into the third system for agitation in the main and oil-purifying still. Reboiler condensate is returned to the boiler feedwater tank, and condensate from the accumulators and stills is clarified in a treating system before returning to the boiler feed-water tank. The balance achieved between the steam systems for the turbines and reciprocating pumps with the agitation and stripping steam requires that current from a large electric-power-generation plant, for the camp and utilities, also be used for the remaining centrifugal pumps for operation. Makeup water for the boilers is produced in an evaporator. Raw water makeup passes through a deaerating heater to the evaporator for chemical treatment. The evaporator is operated with 300 psig steam in the tubes, which evaporates 3000 pounds per hour of raw water in the shell for the 125-psig steam system. The fractionating columns' reboilers require 13,550 pounds per hour of 125-psig steam.

Plant steam is supplied by three 30,000-pound-per-hour bent-tube integral furnace boilers, two of which are capable of supplying the plant's requirements, with the third serving as a standby unit. The boilers operate at 300 psig pressure and a temperature of 500 °F., and have a total heat input of 46,000,000 Btu per hour on 1140 Btu fuel gas. Each boiler is equipped with a 6.5-foot induced-draft fan driven by



Processing Towers

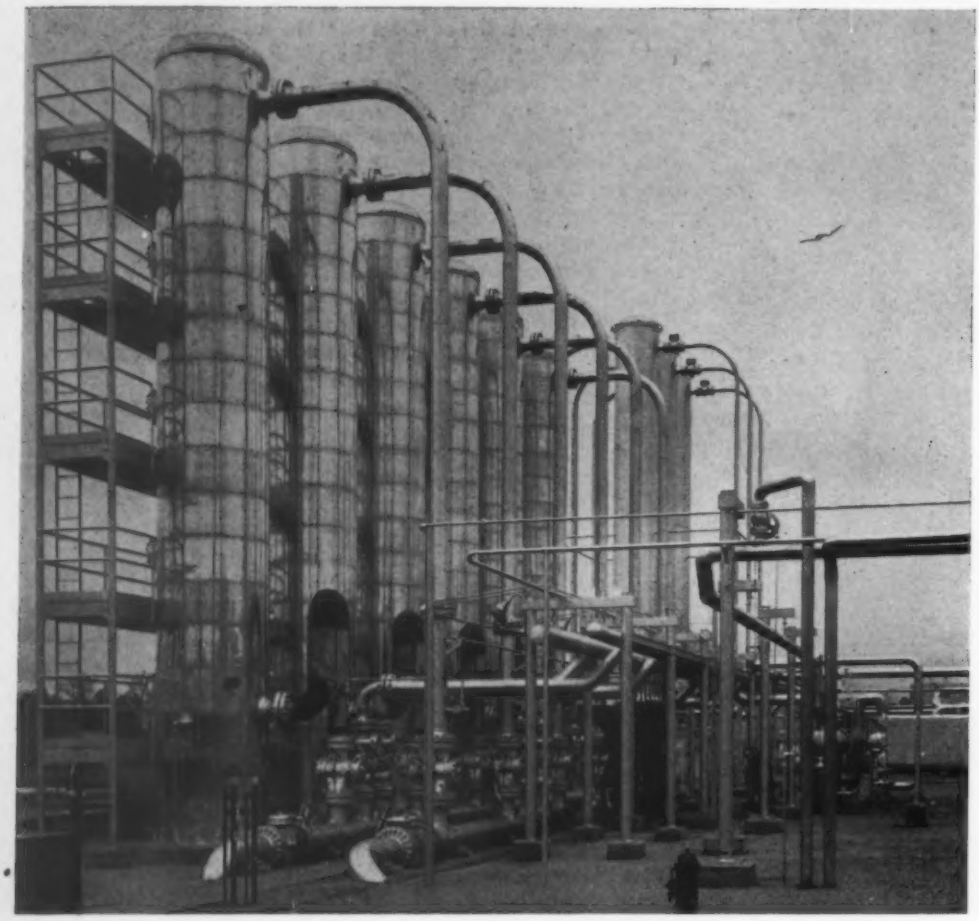
In the foreground, left to right, are high-pressure reabsorber, low-pressure reabsorber, primary still, main still, condensate still and oil purifying still. Beyond the pump house is the de-ethanizer, No. 1 fractionator, debutanizer, depropanizer, and kerosine fractionator.

a 15-horsepower motor supplying total draft requirements of 48,000 pounds per hour.

Absorption

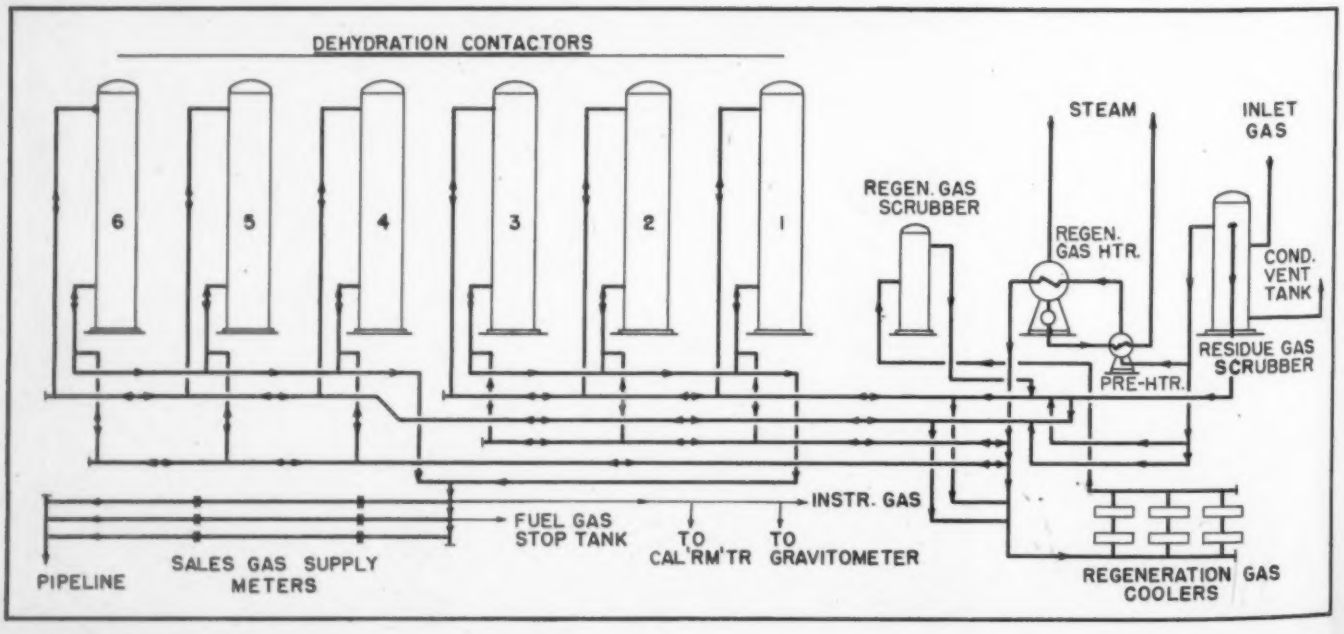
The absorption system of the plant consists of three high-pressure, 24-tray absorbers, 5.5 feet by 50.5 feet, operated at 1250 psig, supplied with lean oil by two six-stage centrifugal pumps, capable of pumping 700 gpm at 1300 psig discharge pressure, driven by 875-horsepower multistage condensing turbines, requiring 8900 pounds per hour of 300-pound steam. There are two reabsorbers in the system. One is a 20-tray tower, 2 feet by 41 feet, operated at 570 psig, which receives its supply of lean oil from the stream to the high-pressure absorbers, and the other is a 20-tray tower, 3 feet by 41 feet, operated at 250 psig and supplied with lean oil from a two-stage centrifugal pump capable of 148 gpm at 250 psig discharge pressure. Two fat-oil vent tanks are used which operate at 570 and 250 psig, respectively.

Inlet gas from the field system (shown on the



Dehydration Contactors

Three of the contactors are on stream while the remaining three are being regenerated. One of the emergency plant-shutdown stations can be seen in front of the first contactor. A flow diagram of the contactors is shown below.



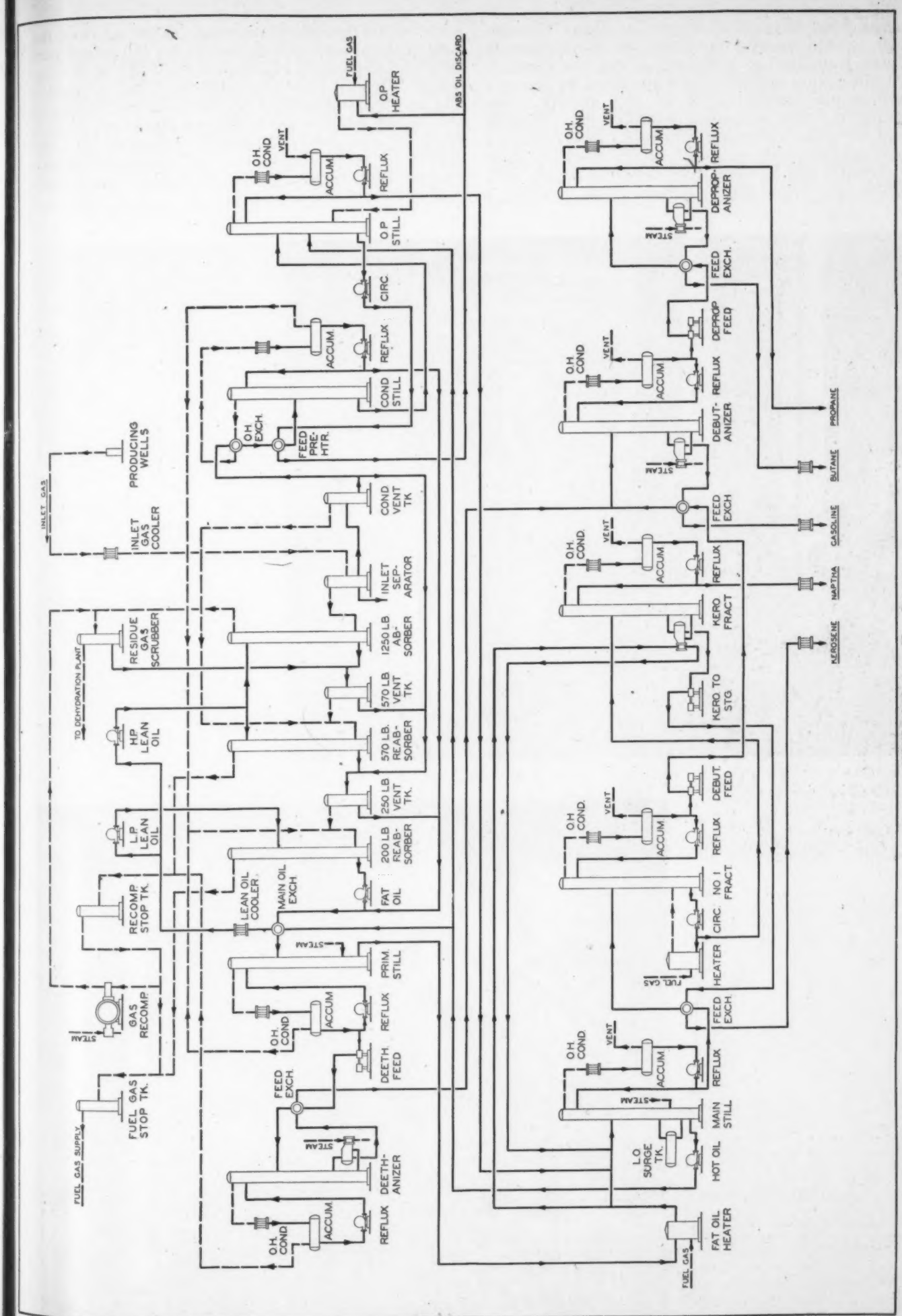


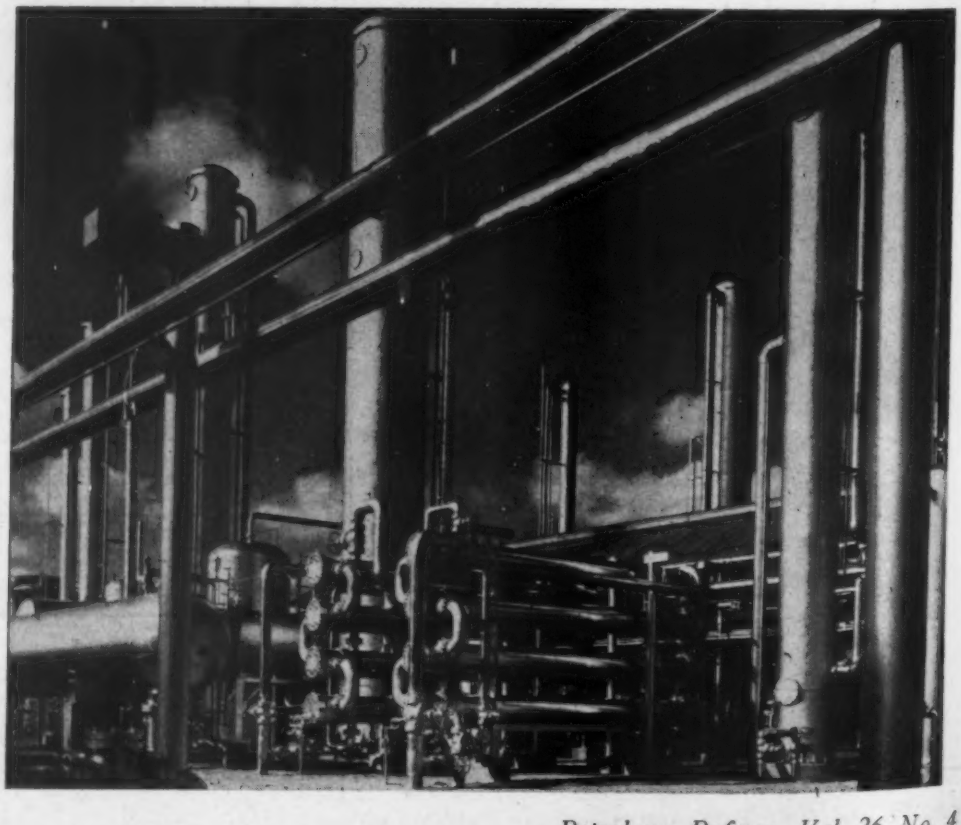
FIGURE 1. Process Flow Diagram

Courtesy Stearns-Roger Manufacturing Company

process flow diagram of Figure 1) flows through coolers and a master separator, where pressure is reduced from 1800 to 1250 psig, to the absorbers. A high absorption efficiency is maintained by using a low-molecular-weight oil of 40 API gravity. Condensate from the separator is flashed in a 575-psig and a 250-psig condensate vent tanks before heating in exchangers with overhead vapors from the condensate still and bottoms from the oil-purifying still. Residue gas from the absorbers flows through a scrubber to the dehydration unit. Rich oil from the base of the absorbers is flashed in the 570-pound vent tank and the vapors charged to the 570-pound reabsorber. Rich oil from the reabsorbed, the 570-pound and condensate vent tanks is flashed in the 250-pound vent tanks. Residue gas is used for plant fuel. Liquids from the vent tank, 200-pound reabsorber, and the condensate-still accumulator are charged through heat exchangers with the hot lean oil to the primary still operating at 215 psig.

Process Pumps

The high-pressure six-stage centrifugal lean-oil pumps, driven by multi-stage condensing steam turbines, capable of delivering 700 gallons per minute at 1300 pounds pressure to the three absorbers and a reabsorber, are shown. The vacuum condensers are mounted on the operating platform above the pumps.

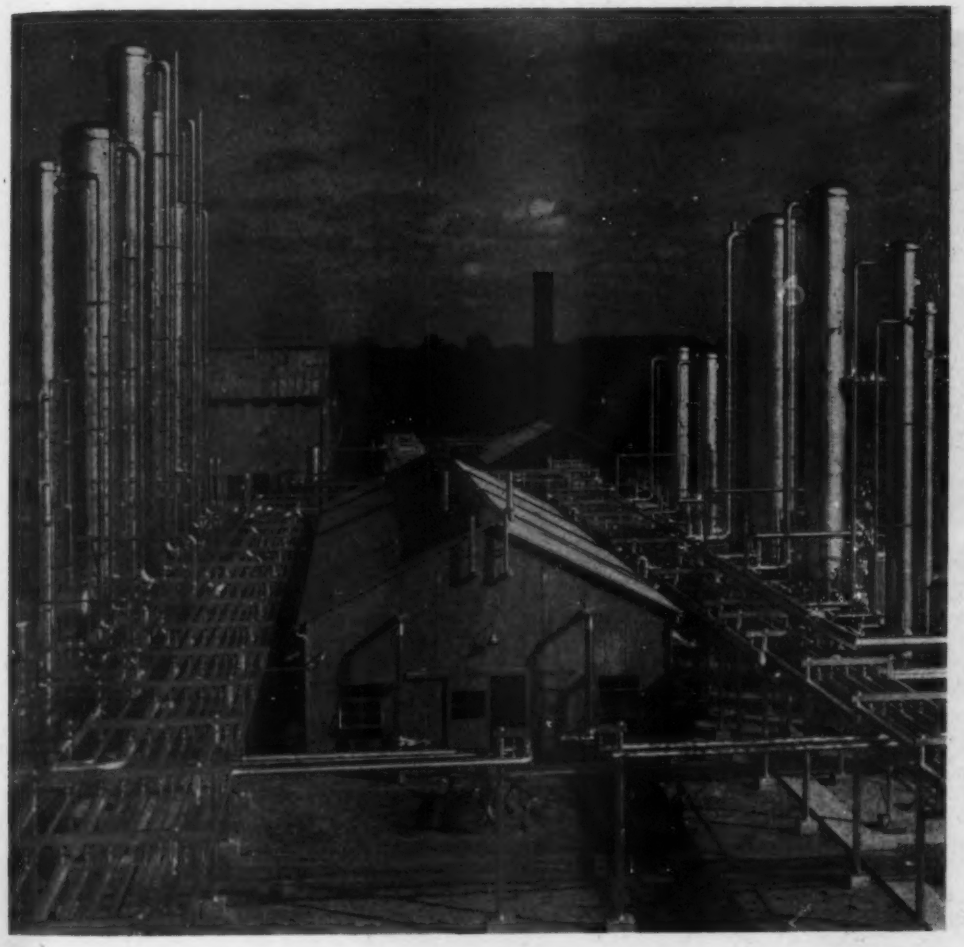


Heat Exchangers

Exchangers for heating the charge to the condensate still. Heat is supplied from oil-purifying still bottoms in one bank of condensers, and from the condensate-still overhead vapors in the other bank.

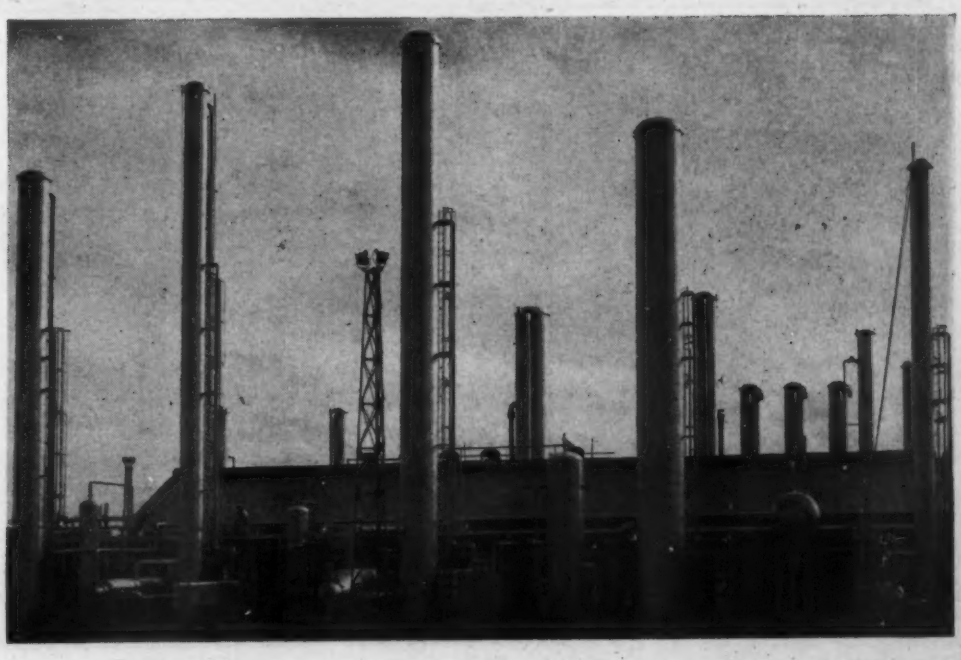
Condensate and Oil Purifying Stills

In the condensate still, which has 16 trays and is 3 feet by 36 feet, the kerosine and lighter hydrocarbons are taken overhead and mixed with the feed to the primary still. The condensate still requires 320 pounds per hour of 300-pound steam. Material from the base of the still is charged to the oil-purifying still, a 16-tray tower, 3 feet by 35 feet, which uses 900 pounds per hour of 15-pound exhaust steam for operation. Absorption oil is produced overhead from the still and is sent to storage or to plant makeup. Approximately 60 gpm of the oil from the base of the still is circulated by a single-stage centrifugal pump, electrically driven, through an oil-purifying heater, and returned as vapor to the still. The circulated oil is heated from 400 to 610° F. in the direct-fired heater which has a radiation section only. The six-burner furnace has a heat load of 3,730,000 Btu per hour and a capacity of 25,500 pounds per hour of 0.85 specific gravity, 240-molecular-weight heavy oil. Remainder



Process Area

View of the plant from the cooling tower, showing the arrangement of towers and piping on each side of the pump building. The boiler house and main heater for the rich oil are in the background.



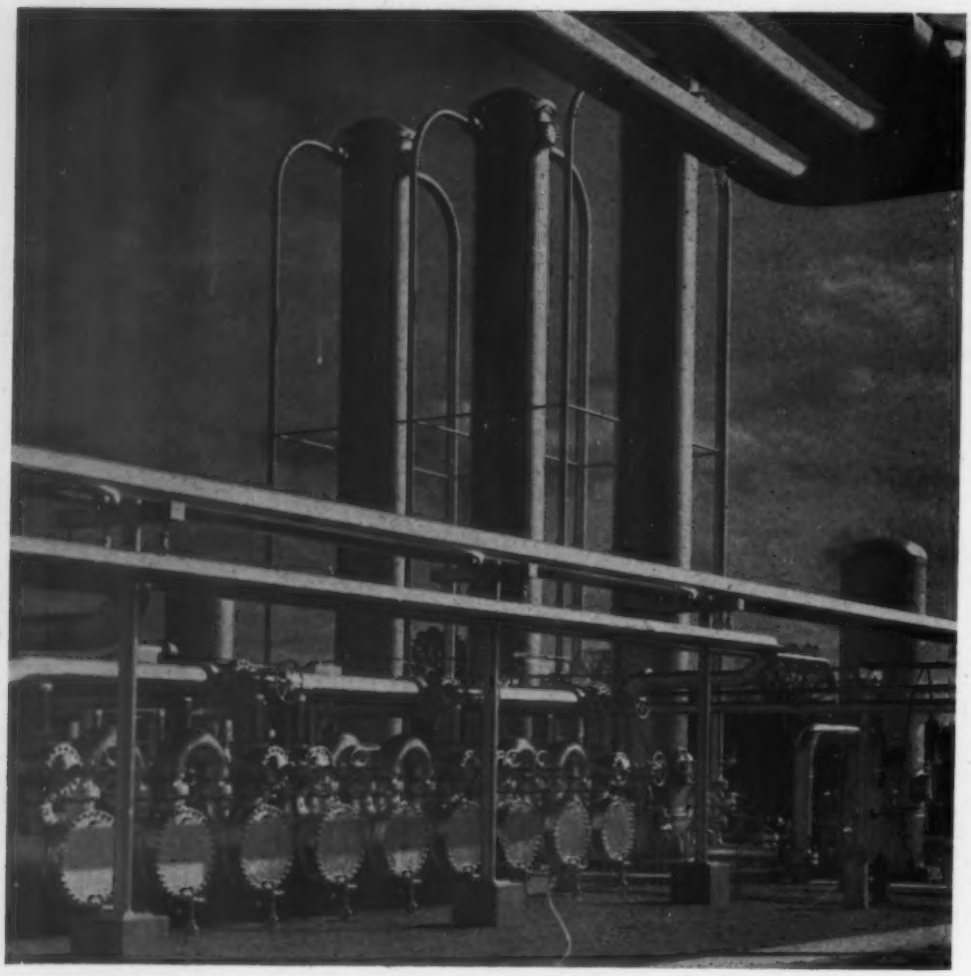
Towers

Kerosine fractionator, depropanizer, debutanizer, No. 1 fractionator, and de-ethanizer columns.

of the bottoms go to heavy-oil storage or the slop tank. In addition to making absorption oil from the condensate charge to plant, the still continuously conditions the oil in use. A small quantity of oil from the stream picked up by the hot-lean-oil pump from the main still is charged to the oil-purifying still.

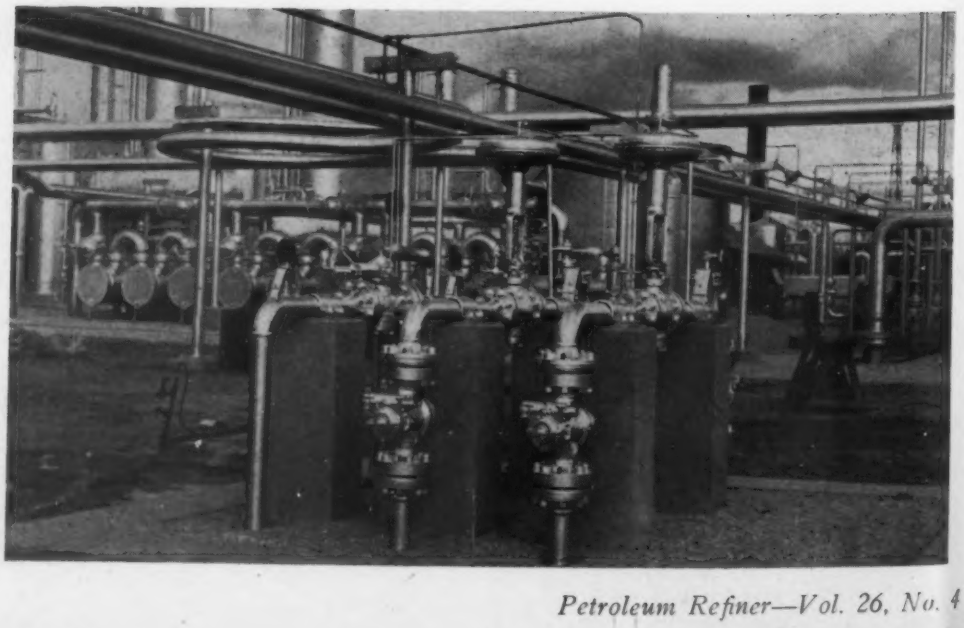
Stripping and De-ethanizing

The primary still used 4940 pounds per hour of 300-pound steam to effect a rough cut of the rich-oil charge, and produces propane and heavier for charging to the de-ethanizer. This column is 6.5 feet in diameter and 53 feet in height, with 22 trays. Rich oil from the base of the primary still is heated from 450 to 565° F. (vaporizing 6100 pounds per hour of 123-molecular-weight hydrocarbons) for charging to the main still, in a pair of direct-fired high-efficiency convection-section heaters set back to back. The furnaces have a total heat load of 30,600,000 Btu per



Heat Exchangers

Main lean oil-rich oil heat-exchanger bank in the foreground, with the three 1250-pound absorbers in the background. The master separator for the inlet gas stream is at the right of the absorber.



Inlet Gas Regulators

Gas enters the plant at 1800 pounds and is reduced to 1250 psig before entering the absorbers by the inlet gas regulators shown.

hour and a capacity of 363,100 pounds per hour of 0.81 specific gravity, 226-molecular-weight fat oil.

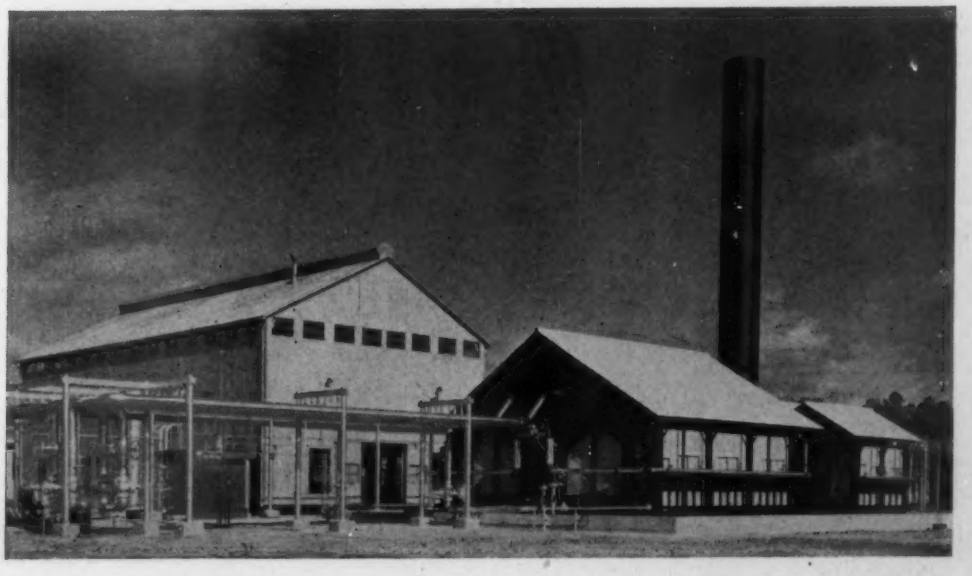
In the 30-tray de-ethanizer, 2.5 feet by 55 feet, ethane is taken overhead from the tower to the recompressor suction, then to plant fuel or the dehydration unit. Propane and heavier from the de-ethanizer reboiler is combined with the debutanizer feed stream from the No. 1 fractionator. The recompressor also is used to compress gases from the high-pressure reabsorber. It is a horizontal opposed steam driven type, and compresses about 4,000,000 cubic feet of gas from 475 to 1225 psig in a single stage.

Fractionation

Fat-oil from the heater to the main still is used to heat the kerosine fractionator reboiler. In addition, a small quantity of lean oil from the oil-purifying still is charged to the still with the fat oil. Kerosine and lighter products are taken overhead from the still, heated by exchange with kerosine fractionator bot-

Steam Generation

Three 30,000-pound-per-hour integral furnace boilers supply steam for plant operation at 300 pounds and 500° F. Total heat input is 46,000,000 Btu per hour on 1140-Btu fuel gas.



Boiler House

Housed here are the three 30,000-pounds-per-hour boilers, the main rich-oil heater, and the oil purifying still heater.

toms, and then charged to the No. 1 fractionator for gasoline production. Lean absorption oil from the base of the main still is pumped through the main oil exchangers and coolers to the feed pump for the absorbers and reabsorber. About 5800 pounds per hour of 15-pound exhaust steam is used in the main still.

The end-point of the gasoline produce overhead from the 30-tray No. 1 fractionator, which is 6 feet by 60.5 feet, can be controlled from 250 to 400° F. A centrifugal pump picks up the kerosine and naphtha from the base of the fractionator and circulates a portion of the stream through the fractionating heater and pumps the remainder to the kerosine fractionator for separation. The kerosine fractionator has 30 trays and is 4 feet by 60.5 feet. The heater is a direct-fired type with a high-efficiency convection section utilizing 8 burners, and has a total heat load of 7,280,000 Btu per hour. Its capacity is 68,000 pounds per hour of 0.79 specific gravity, 187-molecular-weight oil, and vaporization 51,000 pounds per hour. The oil is heated from 450 to 495° F. and returned to the fractionator. Gasoline from the fractionator is pumped through a feed exchanger and combined with the bottom product stream from the de-ethanizer for charging to the debutanizer.

Finished debutanized gasoline from the base of the debutanizer is cooled and sent to storage, and the propane and butanes produced overhead are charged to the depropanizer for separation into propane overhead and butanes from the tower reboiler. There are 40 trays in the debutanizer and in the depropanizer, which are 5 feet by 75.5 feet and 3 feet by 71 feet, respectively.

The plant has a total storage capacity of 7,264,000 gallons for all products, including kerosine, naphtha, gasoline, butane, propane, LPG, and fuel oil. Gasoline produced for motor fuel is blended to the desired octane rating in an ethyl blending unit in the plant. The products are shipped by railroad tank cars and trucks.

Dehydration

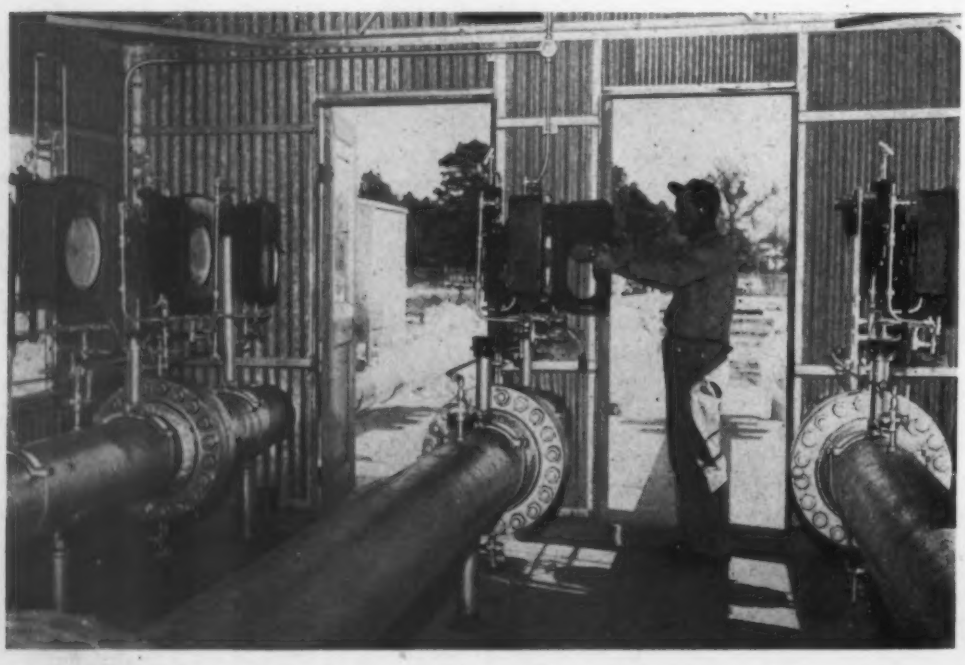
Residue gas from the absorbers flows through a scrubber to the dehydration unit of the plant, Figure 2. The six contactors in the unit, are operated with three contactors on stream while three are being regenerated under full line pressure. The contactors are 6.5 feet in diameter by 32.5 feet in height. Operation is on a 24-hour time cycle. Gas enters the top of the contactors for dehydration for 12 hours. Regeneration gas is taken from the scrubber and heated by steam (8975 pounds per hour of 300-pound steam) in an exchanger to approximately 400° F., then enters the outlet header of the saturated contactors for six hours. The hot wet gas is cooled in the cooling tower, the water removed in a separator, and the gas returned to the inlet header of the contactors. This method of reversing the flow of gas during each cycle for regeneration, and cooling prevents the contactors from making wet gas when first returned to the dehydration stream.

Three meter runs are used in the dehydrated sales gas system, one of which is an auxiliary run, and permits the operator to switch the flow from a meter run to make orifice meter repairs or inspect the orifice plate.

The coils for the contactor cooling gas are in the induced-draft double-flow tower, which is 63 feet wide, 121 feet long, and 35 feet high, with open bent-tube sections and a 10-foot coil shed which is used for all cooling in the plant. The towers have five double cells, each with 8 bays, and five 184-inch three-bladed fans driven at 200 rpm by 50-horsepower electric motors. The heat load represented by water cooling is 107,000,000 Btu per hour, and including evaporative cooling in the coil shed is 110,200,000 Btu per hour. The water capacity at 100° F. is 13,400 gpm, hot water temperature 100° F., and cold water 84° F. Wet-bulb temperature is 79° F.

Emergency Shutdown

The plant can be shut down by a dual system from six emergency stations located throughout the plant. The stations operate piston valves and regulators on one system to stop operation of the plant, except for the boilers. The second system shuts down the boilers and the plant. Inlet and outlet gas is shut down, absorbers and contactors are vented to atmosphere, boiler fuel is cut off, high-pressure pumps are stopped, and snuffing steam is turned into the direct-fired heaters.



Meters

Utilizing two meter runs with the third as an auxiliary, permits the operator to check orifice plates or repair the meters.

Motor Fuel Trends in Relation to Antiknock Requirements

T. R. RISK and JANE F. JORDAN
Engineering Laboratories, Ethyl Corporation

OVER the years, the petroleum industry usually has been capable of producing fuels whose antiknock quality was better than required by most of the current engines. That is, the industry at any given time has been capable of rapidly translating technical knowledge into practice when necessary. It demonstrated this ability to an amazing extent in its rapid conversion to the production of 100-octane aviation fuel. Owing in part to its war experience, the oil industry today should be farther ahead of the automotive industry than ever. It is interesting, therefore, to begin this survey with the motor-gasoline picture. Since the immediate future is of primary importance, the significant data on which to base any predictions are to be found in what has happened to motor fuel antiknock quality since V-J Day when the industry began to convert its gigantic wartime plant for 100-octane aviation fuel to the manufacture of motor fuel.

Figure 1 shows the trends in the Motor and Research method octane ratings of regular and premium-grade gasolines since the initial lifting of all restrictions on civilian fuel just after V-J Day. The solid and dotted lines represent Motor-method and Research-method ratings, respectively. The heavy lines are the average fuel ratings by the respective methods throughout the United States, while the light lines are the maximum values at any one time.

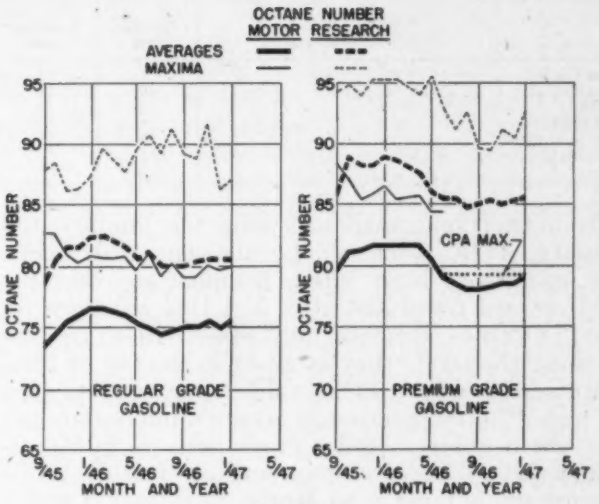


FIGURE 1
Trends in Gasoline Antiknock Levels Since VJ Day.

The latter are values for a single sample, of course, and the maximum Motor and Research ratings at any time were not necessarily obtained on the same sample. These curves show the dip in antiknock levels which fuels took during the latter part of 1946 as a result of the reinstating of government restrictions on tetraethyl lead and maximum octane numbers.

Although average Motor-method data on gasolines are readily available for the years before the war, corresponding Research-method data are very limited

DURING the past fifteen to twenty years, the gasoline provided by the petroleum industry generally has been better from an antiknock standpoint than the design of the majority of the contemporary cars required. This situation has been not only beneficial but necessary to a smooth evolution in motor transportation. It is obvious that the automotive industry could not afford to place many cars on the market requiring fuels better than those generally available. However, there always has been a small minority of engines and vehicles which were designed to take full advantage of the best fuel available. These have been the pace-makers, pointing the way to probable industry-wide developments over the next few years. Fortunately, refining technology has attained a degree of flexibility which has permitted the oil industry to accept the leadership imposed upon it. As the leader, it is important that the oil industry keep abreast of the contemplated changes in automotive design forecast by the pace-makers. Conversely, it is equally important that the automotive industry appreciate the limitations of petroleum refining so that the two may progress together.

The past twenty years have witnessed remarkable progress in the knowledge of the interdependence of fuels and engines. This knowledge is far from complete, but it does permit hazarding some guesses as to the manner in which the present trends in fuels composition and antiknock quality and the present trends in automotive engine design are likely to react on one another. Since the subject is one of tremendous scope, it seems advisable to confine this discussion to the immediate future, that is, to the interval up to about 1950.

The automotive industry, in its effort to keep total production at a high level, has made no essential changes in its 1947 models. It is intimated that few changes are contemplated even for 1948. It seems likely, therefore, that the first particularly different automobiles will be those produced in 1949 or 1950. These vehicles are already past the drawing-board stage. Their exact specifications are closely-guarded trade secrets, but the industry as a whole talks freely of the type of changes which are under discussion.

It is also possible to talk with reasonable accuracy of the trends in gasoline composition and antiknock quality which are likely to obtain up to about 1950. There is the example of some of the 100-octane war plants which have been converted to motor-fuel production on which to draw in predicting the capabalities of the petroleum industry over the next four or five years. To extend the analysis beyond these forseeable future possibilities in either industry would require pure conjecture and reduce whatever practical significance this discussion may have.

Presented before the annual meeting of the Western Petroleum Refiners Association, San Antonio, March 24-26, 1947.

so that it is possible only to estimate the average prewar sensitivity of fuels. Sensitivity is defined as the Research-method rating minus the Motor-method rating. Such data as are available indicate that, on the average, prewar motor fuels were about of the quality shown in Table 1. Similar data for the present from Figure 1 are included for comparison. These figures indicate that the petroleum industry is now able to make fuels which are not only higher in Motor-method octane number but also of greater sensitivity than their prewar predecessors.

TABLE 1

	Pre-War 1941	Post-War 1946
Premium Grade:		
Motor Method	80	82
Sensitivity	6	7
Regular Grade:		
Motor Method	75	77
Sensitivity	5	6

The most recent antiknock data for January and February, 1947, since all government restrictions have once more been lifted, demonstrate that the initial upward trend just after V-J Day has been resumed. Octane numbers and sensitivities are approaching the levels they attained in the fall of 1945. In predicting where these values may go between now and 1950, the maximum octane number data are an excellent guide. Regular grade fuels of 80 Motor-method and 88 Research look entirely feasible while premium grade fuels of 85 Motor, 95 Research rating appear to be equally likely. The existence of a fair proportion of samples over the last year at these levels is rather convincing evidence of the reasonableness of such predictions.

Naturally, refiners who are in a position to make such fuels economically are likely to do so. Other refiners may as a result find themselves at a competitive disadvantage. This latter group may take comfort from the fact that the present passenger-car population probably does not require such fuels and that they can get by without serious complaints from their customers until such time as the automotive industry puts new engines of higher requirement on the market. Actually, it is an interesting fact that, as Hebl and Rendel stated back in 1939, "The so-called octane requirements (of cars) are not a direct function of the design of the cars or their operating conditions, but depend largely on the octane number of the fuel available to them in any given territory."[1]

This fact is illustrated in Figure 2. Surveys of the octane requirements of the passenger-car population current at any time have not been conducted on anything like the controlled, regularized basis of surveys of the laboratory octane numbers of available fuels. Consequently, the data presented in these curves are scattered and by no means infallible. However, they do illustrate the point made by Hebl and Rendel. In this figure, the heavy line of open dots represents the approximate octane number of the secondary reference fuels required to satisfy 80 percent of the cars tested, while the heavy line of solid dots represents the octane number of fuel required, in terms of secondary reference fuels, to satisfy 90 percent of the cars tested. The light lines have been added to indicate the Motor-method level of the premium and regular grade fuels generally available at any particular time.

At first glance, it would appear that the premium fuels available up to 1940 were barely good enough to satisfy 90 percent of the cars on the road. It must be remembered, however, that the octane-requirement curves are actual road requirements. The available fuels, for which only Motor-method octane numbers are shown, had road octane values several octane numbers higher, depending upon their sensitivity. It is a reasonable assumption that the regular-grade gasolines made available by the petroleum industry usually satisfy at least 80 percent of the cars so that it is likely that the road value of the regular-grade gasolines available between 1936 and 1940 approximated rather closely the octane requirement curve for 80 percent of the cars. The same type of reasoning indicates that, in general, premium-grade gasolines have had road antiknock values which were higher than required by 90 percent of the cars.

It will be noted, however, that as octane numbers have climbed, the requirements of the cars also have increased. The 1941 data are particularly interesting in this connection. The curves show that the octane requirements of the cars at this time were approaching premium-gasoline quality to a greater extent than ever before. It should be remembered that the engine manufacturers deliberately retard their distributor curves from maximum power spark advance, especially at low speeds. Thus, when fuel antiknock quality allows it, the motorist can derive benefit from advancing the spark toward maximum power setting. This point is illustrated by the open and solid squares below the octane-requirement lines for the 1941 data. These are the corresponding octane requirements of the same group of cars tested after the basic spark settings had been adjusted to the manufacturer's recommendation. It can be seen from these points that the availability of better gasolines encourages motorists to advance spark timing to take advantage of the fuels. Another factor which influences this tendency toward increased requirement is that with better fuel in the tank, the motorist drives his car longer before deposit accumulation reaches a level where knocking

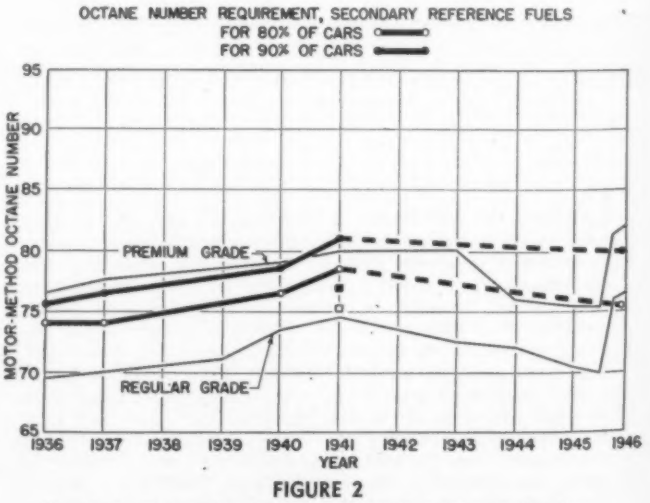


FIGURE 2

Trends in Octane Requirement and Fuel Antiknock Quality.

is bothersome or actual trouble forces him to give his car mechanical attention.

The only octane-requirement data at hand for recent car populations are those shown for the latter part of 1945. These are connected to the 1941 data by the heavy dotted lines. If it is assumed that these dotted lines are reasonable estimates of the requirements of civilian cars during the war period, it is

[1] Hebl, L. E. and T. B. Rendel, J. Soc. Auto. Eng. 44, 210 (1939).

easy to see why the drastic reductions in the quality of civilian fuels necessitated by the emergency brought such an increase in knocking complaints. Even premium-grade fuels were barely able to satisfy 80 percent of the cars. The 10 to 15 percent of the cars which required fuel of better quality than the average premium were subject to considerable difficulty. As a result, spark timing was retarded and in certain cases compression ratios even were decreased to accommodate the cars to the fuels available, with attendant losses in performance and economy, of course. This situation also helps to explain the decided swing toward premium-grade fuels which took place during the war years. This habit, once formed by the motoring public, has persisted so that premium-gasoline sales remain at about 40 percent of the total.

The late 1945 data indicate that fuels are once again in advance of the engine requirements. It will be noted that, for the first time, regular-grade gasolines probably have sufficient antiknock quality on the road to satisfy at least 90 percent of the cars. This is to be expected from the trends toward greater sensitivity illustrated in Figure 1. Premium-grade gasolines are obviously much better than required by any but a few models. However, before refiners who are struggling to meet octane competition take any particular comfort from this momentary advantage, they should remember the facts illustrated by the data from 1936 to 1941. When these better fuels are made available in large quantities in an area, the cars there gradually begin to require them. It is safe to predict that when the higher-octane fuels have been available for six months to a year, that is by the end of 1947, a survey of the octane requirements of cars at that time will show an upward swing. This, of course, is provided there are no further curtailments of the industry's progress.

Trends in Automotive Engine Design

The fuel requirements of the car population from 1947 on naturally will be affected by the influx of new vehicles. The 1947 models now available differ very little from the 1946 cars, and the automotive industry intimates that no drastic changes in design are contemplated for 1948. If this situation does obtain, it is safe to assume that for the next two years fuel requirements largely will follow the pattern of previous years. As better fuels are made available, the motorist will take advantage of the improved operation which they permit by advancing spark settings toward maximum power. The octane requirement of the cars in relation to the Motor-method value of available fuels was indicated in Figure 2. Octane requirement data of this type represent the maximum road antiknock quality required, in terms of secondary reference fuels, regardless of the engine speed at which maximum knock may occur. In order to relate trends in engine design and the antiknock characteristics of fuels, it is necessary to go deeper into the way the engines and fuels react to changes in speed.

The Borderline method of road knock testing is an excellent tool for examining these characteristics. This method utilizes the relationship between ignition timing, engine speed, and fuel knocking tendency. It was adopted by the Coordinating Research Council as a standard road-test procedure for gasolines in 1941 and carries the official CRC Designation F-8A-943. Special equipment is necessary for conducting tests by the Borderline method. The car must be equipped with a fixed distributor, that is, one which will not advance the ignition timing with engine speed, and a satisfactory means for adjusting it from the driver's seat. Likewise, there must be a suitable means for determining the spark advance while the engine is being operated on the road; an electronic or stroboscopic indicator is satisfactory for this purpose. There also should be a tachometer, since engine "revolutions per minute" rather than car "miles per hour" usually is used to indicate speed.

The procedure for determining the borderline curve for a fuel is as follows: The car first is operated for a sufficient time to insure that the test fuel has displaced completely the original tank fuel in the fuel system. Then, the ignition timing is set manually to a relatively low advance, usually 2 to 6 degrees before top dead center. The car is slowed down to as low a speed as possible in high gear, generally about 500 to 660 rpm, and then accelerated with wide-open throttle; the speed at which the knock dies out is noted. This gives one point on the borderline curve—representing the two dimensions, spark advance and knock-die-out speed. The timing then is advanced 2 degrees and the entire procedure repeated to give the second point on the curve. This process is continued until the complete curve has been established for the spark advance and engine-speed range under consideration. Care must be taken to prevent excessive knocking, since this will affect the reproducibility of the knock-die-out point.

Borderline-test data usually are plotted as shown

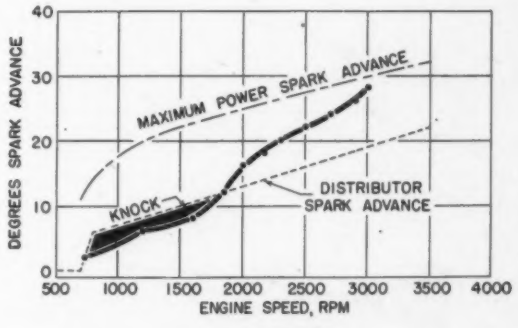


FIGURE 3

Borderline Knock Curve. Method Used to Obtain Curve for Any Fuel.

in Figure 3. The light dotted line represents the distributor spark advance curve for the particular engine in which the tests were conducted. For a fuel not to knock in this car, it must be able to tolerate at least as much spark advance at all speeds as that produced by the distributor-advance mechanism. Wherever the solid line representing the borderline curve for the test fuel falls below the distributor-spark-advance curve, the engine is considered to be knocking. The Borderline curve for this fuel indicates that it will knock in the range from 700 to 1,800 rpm.

In this example, the actual knock-die-out points obtained on the road are indicated by the heavy dots to illustrate the number of points actually obtained in the development of a curve of this type. Each die-out point is checked twice on the road; if there is still some doubt in the operator's mind, additional accelerations are made until the knock-die-out speed for any spark advance has been established definitely. The individual points are not shown on other Borderline curves in this discussion, but all were obtained and recorded in a similar manner.

The Borderline curve obtained in this way will be

indicative only of the antiknock characteristics of the test fuel in the particular type of engine in which the tests were conducted, inasmuch as there is a difference in knocking tendencies between different types of engines. With this test procedure, the valve-in-head engine tends to knock more severely at low speeds than the L-head engine. It is advisable to determine the Borderline curve of a test fuel in duplicate engines of each type. This procedure will permit accurate evaluation of the characteristics of fuels, and will indicate whether antiknock quality is likely to be utilized to best possible advantage.

The octane number of a fuel or the octane requirement of an engine at any engine speed can be obtained by also developing Borderline patterns for a series of reference fuels which completely bracket the spark-advance range of the test fuels and the distributor curve. The Borderline curves for the reference fuels usually are referred to as the reference fuel framework. Test-fuel octane numbers are obtained by interpolation between their Borderline curves and the reference fuel framework at any engine speed. Engine octane requirement at any speed is obtained by interpolation between the distributor-spark-advance curve and the reference-fuel framework. These procedures are illustrated in Figure 4.

FIGURE 4

Estimation of Equivalent Road Octane Number from Borderline Curves.

In most of the conventional cars on the road the maximum octane requirement as determined by the Borderline method occurs in the low-speed range. A typical curve is shown by the solid line in Figure 5. As engine speed increases, the octane requirement of the usual current automotive engine decreases, however, so that at 3000 rpm, or approximately 60 mph, it probably requires a fuel some 8 or 9 octane numbers lower than it did at 800 to 1000 rpm, or up to about 20 mph. The octane numbers shown should be considered only as a qualitative guide. Actual levels will vary greatly, depending upon the type of engine and its compression ratio. In general, the octane-requirement curve for a valve-in-head engine is steeper than that of an L-head engine, demonstrating that the former is relatively more severe at low speeds. However, both types usually have their greatest requirement in the low speed range.

The importance of spark adjustment in altering fuel requirements was stressed in connection with Figure 2. The dotted line in Figure 5 illustrates the manner in which changing basic ignition timing may affect the fuel octane requirement over the speed range. The indicated change of 5 degrees in basic setting has increased the maximum requirement of this engine by about 6 octane numbers. This occurs in the low-speed range where the engine is already most critical. It will be noted, however, that the increase in octane requirement as a result of change of basic setting becomes progressively less as speed increases so that at 3000 rpm the requirement has only risen about 2 octane numbers. It should be remembered that the spark is advanced because fuels are available which will permit it. Motorists tend to take advantage of the gains in performance and economy which better fuels permit them to achieve. In the example in Figure 5, low-speed octane requirement has been increased to a maximum of about 81, in terms of secondary reference fuels. This is the road octane requirement of the engine and is not to be confused with the Motor-method octane number of any particular available fuel. The more sensitive gasolines now being made available have road performances which exceed their Motor-method values by a substantial margin, depending upon the degree of sensitivity and the chemical composition of the fuel. It is reasonably sure that an engine such as the one illustrated here with advanced timing will operate satisfactorily on present-day gasolines.

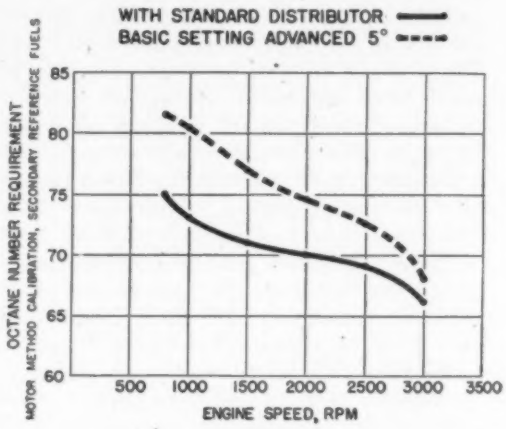


FIGURE 5

Effect of Advanced-Spark Timing on Octane Requirement.

To carry this analysis beyond 1948, it is necessary to make some predictions as to the changes in automotive design which the industry contemplates making in its 1949 to 1950 models. Two design changes which will have substantial effect on engine-fuel requirements and which may be widely adopted by 1950, are higher compression ratios and improved automatic transmissions or torque converters. The automatic transmission has had considerable testing in actual driving since its adoption in two different forms by Chrysler and Oldsmobile in 1938 and 1939, respectively. The general interest of the public in devices which eliminate the conventional gear shift has encouraged all of the manufacturers to devote considerable effort to developing satisfactory automatic transmissions. Even the manufacturers in the low-priced field expect to use such mechanisms.

With this prospect in view, it is interesting to consider how the fuel requirements of the vehicles so equipped may be affected. In Figure 6, the light solid

line indicates the octane requirement over the speed range of a conventional vehicle with a 6.1 to 1 compression ratio engine. It shows the typical high requirement at low engine speed, dropping off as speed is increased. The light dotted line is the estimated fuel requirement curve of the same engine in a vehicle equipped with a fluid torque converter. Since equipment of this type is still in the development

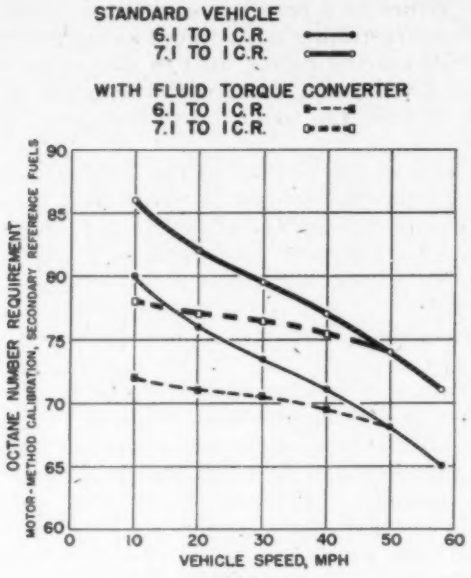


FIGURE 6

Effect of Fluid Torque Converter on Octane Requirement.

stage, no passenger cars embodying the fluid torque converter have been available for actual fuel rating. Data are available, however, on the relation of engine speed to vehicle speed with such installations. The octane-requirement curves depicted here were estimated from the known data on the standard vehicle with proper allowances for the alteration in the vehicle speed to engine speed relation introduced by the fluid torque converter. These curves, therefore, represent only the relative differences in octane requirement which may result from the use of such transmissions. It will be noted that the octane requirement of the vehicle drops noticeably owing to the fact that the fluid torque converter prevents loading the engine at low speeds where the conventional engine with conventional transmission is most critical. The octane requirement of such a vehicle is still greatest at low speeds, however, but in this case the decrease in requirement with increasing speed is slight. In other words, fluid torque converters tend to produce vehicles whose fuel requirements are lower and show less change with speed than those having the same engines with conventional transmission.

The fluid torque converter, however, decreases fuel economy, owing to slippage. Some of the automotive engineers, who have been working on new transmissions, are of the opinion that the use of fluid torque converters will have to be accompanied by increased compression ratios in order to retain fuel economy at an acceptable level. The information at hand indicates that in most instances an increase of approximately one ratio will be necessary to accomplish this. On Figure 6, the heavy solid line shows the octane requirement curve of the standard vehicle equipped with a 7.1 to 1 compression ratio engine while the heavy dotted line indicates the estimated fuel requirements of a vehicle of this same compression ratio equipped with a fluid torque converter. Combining increased compression ratio and fluid torque converter brings the octane requirement at low speeds back to approximately the level of the original vehicle with an engine of lower ratio and conventional transmission. However, the shape of the curve is now such that octane requirement remains fairly constant over the speed range. In other words, this trend in engine and vehicle design will tend to increase high-speed octane requirement while maintaining the critical low speed requirement at approximately the customary levels. Since it has not been possible to check these predictions in cars on the road, they do, of course, represent only a rationalization of the situation as it appears from the available information on the characteristics of the fluid torque converter.

Such trends are of great import to the petroleum industry. They stress the fact that while it is still of primary importance to build fuels with good low-speed antiknock characteristics, it is also necessary to see that the fuels to be used in such vehicles maintain their antiknock quality over the speed range. Fuels which knock at low speeds in vehicles of the type depicted here are liable to continue knocking over the greater portion of the speed range. Such persistent knocking may be far more damaging than the type of knock to which the industry is accustomed from past experience, which generally obtains briefly up to about 20 mph and then vanishes.

Motor-Fuel Antiknock Characteristics on the Road

In view of the probable octane requirements throughout the speed range of cars which will be available up to 1950, it is now of interest to examine the road antiknock characteristics of the fuels which are likely to be marketed during this period. It should be apparent from the previous discussion that the problem is not one of the basic octane number of the fuels, but of their effective antiknock quality on the road, and particularly the variation of inherent antiknock quality with engine speed. All of the fuels discussed in the following section were rated on the road in conventional type vehicles.

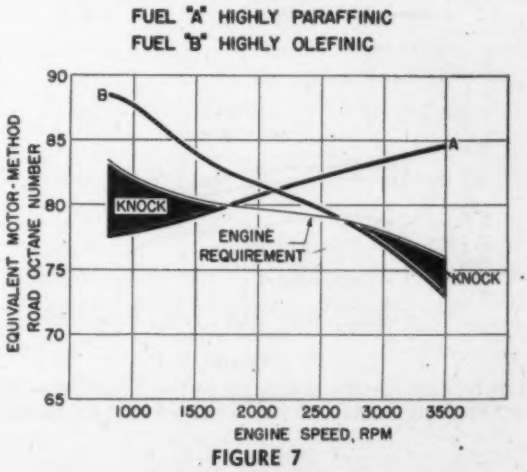


FIGURE 7

Borderline Knock Curves.

The usefulness of the Borderline method for studying the variation of fuel antiknock quality with engine speed is demonstrated in Figure 7. Fuels A and B are distinctly different in chemical composition, one being highly paraffinic, like a straight run gasoline, the other being highly olefinic, like a cracked gasoline. By the older methods of knock rating gaso-

lines on the road, both fuels are assigned the same road octane number. The older Uniontown method simply brackets the fuels in terms of maximum knock intensity between reference fuels of known octane number which knock just slightly less and slightly more than the test fuel. This method pays no attention to the speed at which maximum knock occurs, and consequently, gives no information as to the antiknock properties of the fuels in the speed range in which there is no knock. The Borderline patterns

FUEL NO.	TEL CC/GAL	OCTANE NUMBER MOTOR	RESEARCH
A	0.0	66.0	69.5
B	0.5	70.0	75.0
C	1.5	76.0	81.5
D	3.0	79.5	85.5

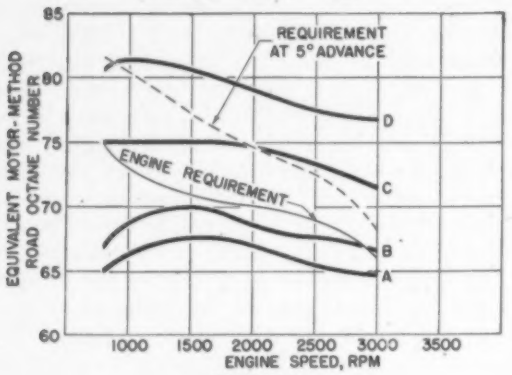


FIGURE 8

Regular-Grade Gasoline, 70 Percent Cracked, 30 Percent Straightrun.

of these fuels, however, indicate clearly the differences between them. The highly paraffinic fuel, A, knocks severely in the low-speed range from 800 to about 1800 rpm but is much better than the engine requires at high speeds. The highly olefinic fuel, B, has excellent low-speed antiknock characteristics but knocks at high speeds above 2800 rpm.

Actually, most motor fuels are blends of these two extremes. In Figure 8 are shown the Borderline

	FUEL	OCTANE NUMBER MOTOR	RESEARCH
A	REGULAR GRADE BASE	76.0	81.5
B	+N-PENTANE	72.0	79.5
C	+ISOPENTANE	76.5	83.0
D	+NEOHEXANE	76.5	85.5

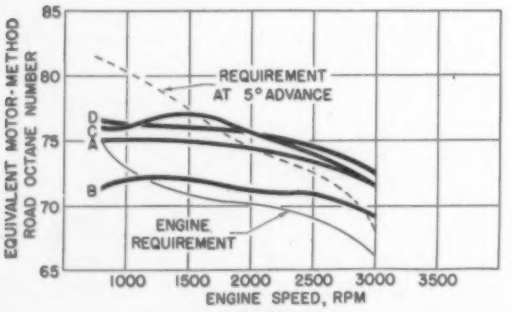


FIGURE 9

Effect of Type of Light Blending Agent on Low-Speed Antiknock Quality. All Fuels Contain 1.5 c.c. Tetraethyl Lead Per Gallon.

curves of a typical commercial regular grade gasoline, consisting of about 70 percent cracked gasoline and 30 percent straightrun gasoline, clear, and with the addition of three concentrations of tetraethyl lead. To satisfy the engine in which these tests were conducted requires the addition of 1.5 c. c. of lead to this base stock, giving a fuel of about 76-Motor-method, 81.5-Research-method octane rating. On the road, this blend rates only 75 according to the borderline data, but this is satisfactory for the engine which, incidentally, happens to be an L-head type of 7.5 to 1 compression ratio. However, the important thing is not the equivalent road octane rating of this fuel at the speed of maximum knock, but the shape of the fuel's Borderline curve. It will be noted that the fuel pattern tends to be quite flat over the lower part of the speed range. The engine requirement curve, on the other hand, is steepest in this portion of the speed range. Thus, when the requirement of the engine increases, either as a result of carbon accumulation, or through spark-timing adjustment as suggested by the dotted line on the figure, fuel of this type begins to knock at low speeds. Even with the addition of a full 3.0 c. c. of lead, the tendency to low-speed knock remains.

This is a characteristic of the majority of typical prewar motor-fuel stocks. A great deal of work has been directed toward determining the factors which influence the inherent road antiknock characteristics of fuels, and especially their low-speed performance since at present this is the range in which knocking is most critical. Experience has indicated that the chemical composition of the lowest-boiling fractions of the fuel is of primary significance in this respect. This fact is demonstrated in Figures 9 and 10. The series of fuels shown here was prepared by stripping the regular-grade base stock just discussed through its C_5 fraction, the material removed constituting about 19 percent of the total fuel. To 81 parts of the stripped base were then added 19 parts of each of the light blending agents to be tested.

The fuels were rated in the laboratory and on the road, clear, and with three concentrations of tetraethyl lead. For simplicity only the blends containing 1.5 c. c. of tetraethyl lead per gallon are shown on these curves. It will be recalled that this is the lead concentration required in the original base stock to just satisfy the antiknock requirements of the test engine. The light paraffinic blending agents shown

	FUEL	OCTANE NUMBER MOTOR	RESEARCH
A	REGULAR GRADE BASE	76.0	81.5
B	+PENTENE-2	76.5	86.5
C	+TRIMETHYLETHYLENE	77.0	89.0
D	+CYCLOPENTANE	78.0	84.5

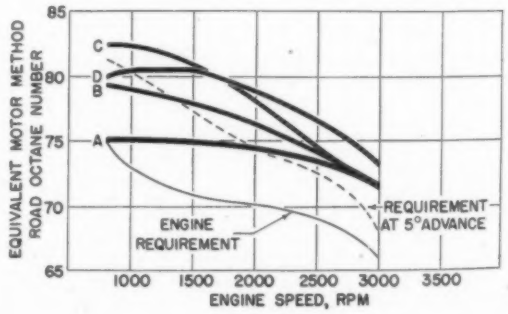


FIGURE 10

Effect of Type of Light Blending Agent on Low-Speed Antiknock Quality. All Fuels Contain 1.5 c.c. Tetraethyl Lead Per Gallon.

in Figure 9 effected changes in the road antiknock characteristics of the base fuel which were roughly in line with the indicated changes in laboratory octane number. Thus, the substitution of n-pentane decreased the Motor-method rating of the base by about 4 octane numbers, and there was a corresponding derating on the road throughout the whole speed range. Iso-pentane and neohexane, on the other hand, improved both the Motor and Research ratings somewhat, and the road ratings of these blends were

slightly better than that of the base fuel. In all cases, however, the Borderline curves of the blends parallel the Borderline pattern of the base fuel, so that when the octane requirement of the engine increases as suggested by the dotted line, even the iso-pentane and neohexane blends will knock up to 1500 to 1800 rpm.

Improvement in Low-Speed Performance

In contrast to the light paraffins, the substitution of the olefinic blending agents shown in Figure 10 made a substantial improvement in low-speed antiknock performance of the base fuel. The normal olefin, pentene-2, was less effective than the highly-branched trimethylethylene, of course, in keeping with the general superiority of branched-chain compounds over their straight-chain isomers. As a result of this improvement in low-speed performance, such fuels are not likely to give trouble when the requirement of the engine increases as suggested by the dotted line. The trimethylethylene blend is even better than the engine requires at this level, a fact which

ENGINE	FUEL	TEL CC/GAL	OCTANE NUMBER MOTOR	OCTANE NUMBER RESEARCH	ENGINE
L-HEAD 1942 8.5 TO 1 C.R. 8 CYL.	A	0.0	82.1	95.4	VALVE-IN-HEAD 1946 8.0 TO 1 C.R. 8 CYL.
	B	0.5	82.9	96.9	
	C	1.5	83.6	98.7	
	D	3.0	84.4	100.0	

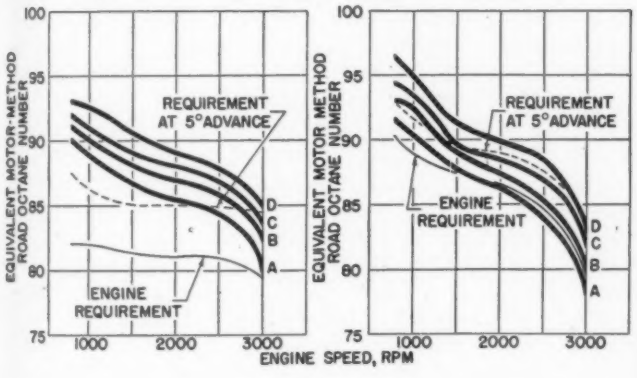


FIGURE 11
High Sensitivity Gasoline No. 1, Made by Catalytic Cracking.

could not have been deduced from its Motor-method rating which is only 0.5 to 1.0 octane number better than that of the other blends.

The curve for the blend containing cyclopentane is included with the olefins as a matter of interest. Although cyclopentane is a naphthene and might have been expected to act more or less as the paraffins do, it actually has the same type of effect on low-speed performance as an olefin.

Consideration of the curves presented in Figures 9 and 10 will suggest that good low-speed performance is a function of fuel sensitivity. Concentrating olefins in the light portion of the boiling range does increase sensitivity as the Motor and Research octane numbers of these blends indicate. The knowledge that higher Research ratings usually mean that there are more olefins in the fuel, and thus, that the low-speed performance is better has led many refiners to control and/or specify their products on the basis of Research octane number. Such a criterion may be misleading if used alone, especially in view of the changes in the octane requirements of vehicles with respect to speed which have been discussed earlier in this paper.

Figure 11 presents the Borderline curves for a high-sensitivity gasoline, No. 1, made by catalytic cracking, clear and with the addition of 0.5, 1.5, and 3.0 c. c. of lead per gallon. Comparable data obtained both in an L-head and valve-in-head engine are shown. It will be noted that the clear fuel with an 82.1 Motor-method octane number and a sensitivity of about 13 satisfies the low-speed requirements of both engines, but is not adequate at high speeds in the valve-in-head, and is barely good enough for the L-head engine in this speed range. Considering that

ENGINE	FUEL	TEL CC/GAL	OCTANE NUMBER MOTOR	OCTANE NUMBER RESEARCH	ENGINE
L-HEAD 1942 8.5 TO 1 C.R. 8 CYL.	A	0.0	79.6	88.8	VALVE-IN-HEAD 1946 8.0 TO 1 C.R. 8 CYL.
	B	0.5	82.2	91.9	
	C	1.5	85.2	94.6	
	D	3.0	87.9	96.7	

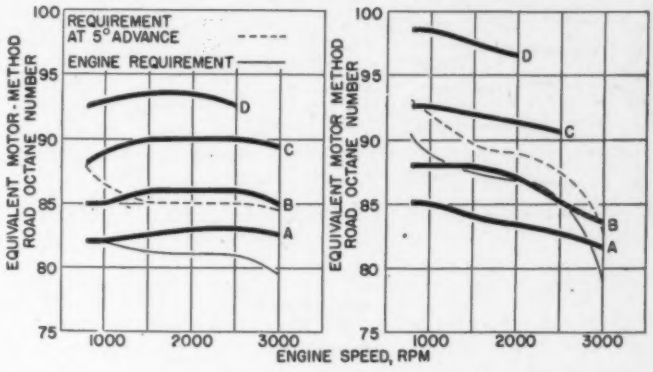


FIGURE 12
High Sensitivity Gasoline No. 2, Made by Catalytic Cracking.

engine requirements may increase, as the vehicles are driven, in somewhat the proportions indicated by the dotted line for 5-degree advance in basic spark timing, it is apparent that this tendency toward high-speed knock in fuels of this type may present serious problems. Even with the addition of a full 3.0 c. c. of tetraethyl lead, such a fuel is barely satisfactory at high speeds when the requirement has increased. This possible situation would never be suspected from a consideration of the laboratory octane numbers of the fuel nor from a typical road octane number determination by the CFR Uniontown method. Furthermore, such fuels are likely to give real trouble at high speeds when vehicles with fluid torque converters and higher-compression-ratio engines become generally available since, as the previous discussion has indicated, the high-speed antiknock requirements of such vehicles tend to be substantially above those of conventional cars.

Not all high-sensitivity fuels are of this type, of course. Figure 12 presents similar Borderline curves at four lead concentrations for catalytically-cracked fuel, No. 2, which has excellent high speed antiknock characteristics. Although the Borderline curves are quite flat in the low-speed range, the response of this fuel to tetraethyl lead additions, at low speeds as well as high, tends to compensate for the inherent weakness shown by the clear fuel. With the addition of 1.5 c. c. of lead, such a fuel should be satisfactory over the whole speed range in both the L-head and valve-in-head engines shown here, even if octane requirement should increase as a result of spark-timing changes or the installation of a fluid torque converter coupled with a higher-compression-ratio engine.

Certain types of thermal cracking also give high-sensitivity gasolines which approach the desirable

antiknock properties demonstrated by fuel No. 2. Figure 13 presents the Borderline curves for high-sensitivity gasoline No. 3 made by thermal cracking. For comparison with the previous fuels, the curves are shown as determined in the same two engines, although this gasoline is 5 to 7 octane numbers lower in clear Motor-method rating. It is hardly fair, therefore, to consider its antiknock properties in relation

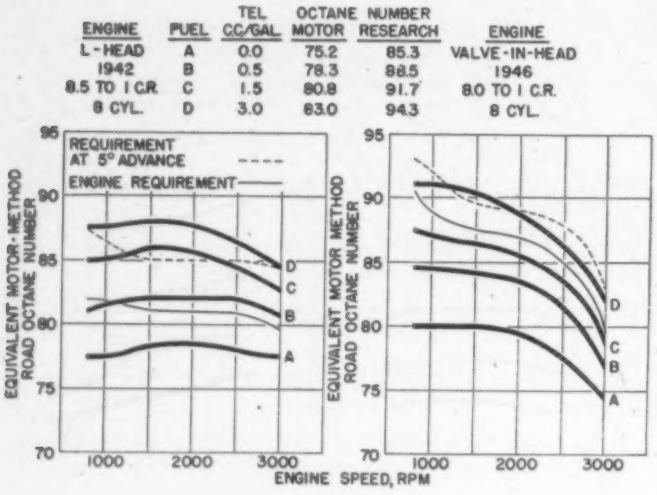


FIGURE 13
High Sensitivity Gasoline No. 3, Made by Thermal Cracking.

to the requirements of the engines. If the shape of the Borderline curves rather than their octane levels is examined, however, it will be seen that fuel No. 3 tends to approach the characteristics of fuel No. 2 on the road. The borderline patterns of the clear fuel show it is inherently somewhat weak at both low and high speeds, but its good road lead susceptibility compensates to a large extent for these inherent defects. With the addition of 3.0 c. c. of lead, this fuel will satisfy the requirements of the two types of engines when essentially free from combustion-chamber deposits, and is nearly good enough to withstand increases in engine requirement due to advanced spark timing.

Some interesting indications can be derived from a consideration of the data shown in Table 2 for the leaded blends of these three fuels which appear to best satisfy the projected requirements of the test engines. It is an interesting point that the blends required to satisfy the engines have essentially the same Motor-method octane number, about 85. A certain degree of sensitivity obviously is necessary in order to hold this Motor-method level on the road. It

TABLE 2
Summary of Antiknock Data

	HIGH SENSITIVITY GASOLINE		
	No. 1	No. 2	No. 3
Tetraethyl Lead, cc./Gallon	3.0	1.5	3.0
Octane Number:			
Motor Method	84.4	85.2	83.0
Research Method	100.0	96.7	94.3
Equivalent Road:			
1000 r.p.m., L-Head	92.5	89.0	87.5
1000 r.p.m., Valve-in-Head	95.0	92.5	91.0
2500 r.p.m., L-Head	88.0	90.0	86.5
2500 r.p.m., Valve-in-Head	88.5	90.5	86.5
Laboratory Sensitivity	15.6	11.5	11.3
Laboratory Lead Susceptibility	2.3	8.3	7.8

TABLE 3
Hydrocarbon Type Analysis of Fuels 1, 2, 3

	HIGH SENSITIVITY GASOLINE		
	No. 1	No. 2	No. 3
Light Cut, IBP to 140° F.:			
Percent Olefins	73.0	21.2	43.0
Percent Aromatics	...	...	...
Percent Paraffins	27.0	78.8	57.0
Percent Naphthenes	...	...	...
Percent of Total Fuel	14.3	22.0	21.3
Octane Number, Motor	90.2	91.2	86.7
Octane Number, Research	Over 100	99.5	99.0
Heavy Cut, 248° F. to EP.:			
Percent Olefins	24.0	1.2	20.8
Percent Aromatics	64.2	54.0	36.1
Percent Paraffins	2.0	4.9	5.7
Percent Naphthenes	10.0	39.9	37.9
Percent of Total Fuel	48.8	51.3	39.9
Octane Number, Motor	73.6	77.9	71.0
Octane Number, Research	81.0	87.5	79.3

would appear, however, that if this sensitivity is gained at the expense of a drastic decrease in lead susceptibility, as in fuel No. 1, the advantage of enhanced road rating at low speeds is no guarantee of satisfactory performance over the speed range.

An analysis of the hydrocarbon compositions of these three fuels furnishes better evidence of their relative road antiknock and road lead susceptibility properties. A good deal of experience along these lines has indicated that it is the composition of the lightest and heaviest fractions of the fuel which is of primary significance. Up to the present time, no correlations of road performance with the hydrocarbon type compositions of more specific cuts have proved practicable. In Table 3, therefore, only the analyses of the lightest fraction boiling up to 140° F. and of the heavy portion of the gasoline boiling above 248° F. have been listed.

These data show that fuel No. 1, which has the remarkable low speed performance, has 73 percent olefins in its lightest fraction. Fuel No. 2 which is particularly good at high speeds and has excellent road lead susceptibility has almost no olefins in its high-boiling fractions. Many data are available to substantiate this evidence that low-speed performance improves with increasing olefin content of the lightest fractions, and high-speed performance and road lead susceptibility improve as the olefin content of the heavy fractions is reduced.

Fuel No. 3, which falls between these two limits in road performance, has about 43 percent olefins in the front end and about 20 percent olefins in the heavy end. It seems unlikely that the drop of only 4 percent in heavy olefins shown by fuel No. 3 in comparison to fuel No. 1 could account entirely for the difference in high-speed performance and road lead susceptibility demonstrated. It seems more logical that although the primary thing is to keep the olefin content of the heavy fractions low, it is of secondary importance to see that the preponderance of hydrocarbons in this portion of the boiling range are those which respond well to additions of tetraethyl lead. Generally speaking, the paraffins and naphthenes have the best lead susceptibility, but previous work indicates that the higher-boiling, highly-alkylated aromatics also respond well to lead additions. This combination of factors appears to justify the superiority of fuel No. 3 over fuel No. 1 at high speeds. The latter has a combined paraffin-naphthene content in the heavy fraction of only 12 percent against about 44 percent in the former. There is no indication in these data as to what percentage of the 64 percent heavy aromatics in fuel No. 1 and the 36 percent

heavy aromatics in fuel No. 3 might be of the highly alkylated type having good lead response, but this factor undoubtedly is significant.

Application of Road Performance Characteristics of Fuels to Refinery Operation

Knowledge of the road antiknock characteristics of gasolines in relation to their hydrocarbon compositions can be used advantageously by the refiner not only in determining the most desirable premium-and regular-grade blends to make but also in deciding on the types of new plant equipment to be added. A general approach to the utilization of this knowledge for these purposes can be gained by an analysis of the data presented in Table 4, Parts A and B, which give the hydrocarbon compositions of the IBP-140° F. and the 248° F. to EP fractions of a number of present-day motor-fuel components. A consideration of these data in the light of the previous discussion of the influence of hydrocarbon composition on the road performance of fuels points out some interesting facts about refinery operations.

Most of the smaller refineries and a number of the larger refineries have available for motor-fuel production only straightrun gasoline, mixed-phase thermally-cracked gasoline, and catalytic polymer. The hydrocarbon analyses of typical examples of these components show that blends of these full-boiling-range stocks tend to be quite paraffinic in the light fractions. Straightrun contains almost 100 percent paraffins, and the mixed-phase cracked gasoline contains 65 percent paraffins in this range. The percentage of polymer boiling below 140° F. is so small that it has comparatively little effect in spite of being completely olefinic. As a result, such blends tend to be poor at low speeds on the road. In refineries where vapor-phase thermally-cracked gasoline is available, motor-fuel blends generally show better low-speed performance owing to the fact that this component is generally about 70 percent olefinic in the light fractions.

In regard to high-speed performance and road lead susceptibility, the typical blends of straightrun, mixed-phase cracked, and polymer probably will be satisfactory owing to the highly saturated character of the heavy portions of the first two components which constitute the greatest share of the fuel. The extreme olefinicity of the polymer tends to offset this somewhat but usually does not produce a critical condition. The inclusion of vapor-phase cracked gasoline, however, may throw the balance into the critical range if present in large volume, since this component is highly unsaturated in the heavy fractions.

If vapor-phase cracked gasoline is used instead of mixed-phase, it would be highly desirable also to be able to substitute thermal polymer for catalytic polymer. Thermal polymer, in contrast to catalytic polymer, has very good lead susceptibility, as the hydrocarbon data would indicate. This would help the critical high-speed condition introduced by the vapor-phase gasoline.

Until the war, these components constituted the major portion of all motor-fuel blends. Now, the components shown in Part B of Table 4 are assuming more and more significance. It is important that refiners contemplating the addition of one of the new types of units recognize exactly how the new fuel component will contribute to the road performance of older blending stocks.

Polyforming, which is presently used primarily for reforming heavy gasoline, produces a fuel quite a lot like thermal polymer in hydrocarbon composition. Polyforming is a thermal operation in which the liquid charge is cracked in admixture with C_3 and C_4 gases. Polyformate is somewhat more unsaturated in the front end than thermal polymer, which enhances its low-speed performance. Though not sufficiently olefinic to equal the low-speed performance of vapor-phase cracked gasoline, polyformed gasoline offsets this by having better road lead susceptibility, and usually a higher octane rating. Being primarily a reforming operation, it is not entirely fair to compare polyforming to thermal cracking of heavier stocks.

The three catalytically-cracked gasolines presented in Table 4, Part B, have been chosen to demonstrate the wide variations in hydrocarbon composition, and, therefore, road performance, which are obtainable with this processing method, depending upon the type of charging stock, the type of catalyst, the cracking temperature, and the severity of the operating conditions selected. In view of the previous discussion, the data presented here need little further discussion. It is important, however, that the refiner considering the installation of catalytic cracking do so in full knowledge of the type of catalytically-cracked fuel he needs to augment his present refinery situation. Obviously, the cost of obtaining these different types of fuel by catalytic cracking varies considerably. It is possible that some less-expensive processing method may be available which will give him almost the same result for a smaller investment.

Hydroforming, though a catalytic-cracking operation, produces a far more specific product owing to the type of charging stock and the circulation of free hydrogen. It is almost entirely paraffinic in the front end, and almost entirely aromatic in the heavy fractions. Road data show it to have unusually good road lead susceptibility at all speeds. The hydroformate

TABLE 4-A

Hydrocarbon Type Analyses Typical Motor Fuel Components

	Straight-Run Gasoline	Mixed-Phase Thermal Cracked	Vapor-Phase Thermal Cracked	Catalytic Polymer	Thermal Polymer
Approx. Boiling Range, °F.	105—270	100—415	100—405	115—380	105—370
Light Cut, IBP. to 140° F.:					
Percent Olefins	0.5	36.0	70.0	100.0	34.0
Percent Aromatics	....	....	....	....	....
Percent Paraffins	99.5	64.0	30.0	....	66.0
Percent Naphthenes	....	....	....	....	....
Percent of Total Fuel	20.2	20.7	21.3	2.1	43.2
Heavy Cut, 248° F. to EP.:					
Percent Olefins	1.0*	31.0*	43.0*	83.0	17.0*
Percent Aromatics	8.0	5.0	25.0	17.0	41.0
Percent Paraffins	44.2	9.0	17.0	....	4.0
Percent Naphthenes	47.8	55.0	15.0	....	38.0
Percent of Total Fuel	7.2	53.3	50.7	29.0	16.8

* Cut 235° F. to EP.

TABLE 4-B

Hydrocarbon Type Analyses Typical Motor Fuel Components

	Poly-Formed Gasoline	Catalytic Cracked Gasoline 1	Catalytic Cracked Gasoline 2	Catalytic Cracked Gasoline 3	Hydro Formed Gasoline
Approx. Boiling Range, °F.	100—425	100—400	90—400	95—400	150—365
Light Cut, IBP. to 140° F.:					
Percent Olefins	43.0	73.0	21.2	56.8	7.5
Percent Aromatics	....	....	....	....	....
Percent Paraffins	57.0	27.0	78.8	43.2	84.3
Percent Naphthenes	....	....	....	....	8.3
Percent of Total Fuel	21.3	14.3	22.0	20.8	2.0
Heavy Cut, 248° F. to EP.:					
Percent Olefins	20.8	24.0	1.2	12.2	1.4
Percent Aromatics	36.1	64.2	54.0	46.6	85.1
Percent Paraffins	5.7	2.0	4.9	21.4	7.8
Percent Naphthenes	37.9	10.0	39.9	19.8	5.7
Percent of Total Fuel	39.9	48.8	51.3	37.9	31.7

tested was quite deficient in light fractions, which undoubtedly was at least partially responsible for the inherently poor low-speed performance noted.

It is not the purpose of this paper to lay down any rules for determining which of the newer types of processing refiners should add to their plants. That decision rests on a number of factors in addition to the road-performance characteristics of the products, and is highly individual. One factor which must be weighed carefully is the sales balance between gasoline and distillate fuel. In cases where the distillate demand is rising, the problem of what to do about low-octane straightrun gasoline becomes even more acute. Catalytic cracking, as presently carried out using gasoil charge, is not helpful in solving this problem and operates in direct competition to the market for virgin distillate. In many of the large plants having cat crackers, a great deal of effort is being directed toward using catalytic cracking for processing the heavy, residual stocks which do not enjoy the market value of the distillate cuts. Some sort of reforming appears desirable as a companion process. It would seem that this trend is an important one in view of the predictions that in the near future distillate demands may approach gasoline demand.

The Fuel-Engine Relation

The preceding discussion of the road antiknock characteristics of fuel components as a function of their hydrocarbon compositions, and the relation of these inherent fuel properties to the probable antiknock requirements of vehicles over the next few years has been treated in its simplest terms. It would be a serious omission to conclude this presentation without mentioning the influence of certain engine factors and test-method variables on the data discussed. The fuel-engine relation actually has three aspects:

1. The fuel, including
   a. The inherent antiknock qualities of the different types of hydrocarbons.
   b. The blending characteristics of these hydrocarbons in the individual mixtures.
   c. The effect of tetraethyl lead additions on the finished blends.
2. The engine, including
   a. The distribution of light and heavy fractions in the induction system of the multi-cylinder engine.
   b. The inherent severity of the individual engine.
   c. The method of loading the engine inherent in the vehicle design.
3. The test method, including
   a. The operating conditions under which it is conducted.
   b. The type of reference fuel used.

All of these factors operate simultaneously to determine the characteristics assigned to a given fuel or engine. For example, it has been assumed in presenting the previous data that the fuel requirements of the test engines were fixed along the lines of octane requirement with respect to speed that have been superimposed on each of the fuel patterns. It will be noted that these fuel requirement curves for the test engines have been labelled "Equivalent Motor-Method Road Octane Number-Secondary Reference Fuels." This is important, for the fuel requirements of the engine vary with the type of reference fuel used to define them. The secondary reference fuels, A, C, M, F, etc., which have been used for so many years were made from selected straightrun-gasoline cuts and alkylate. If, however, in place of these reference fuels the primary standards, isooctane and n-heptane, or blends of diisobutylene and n-heptane had been used, the equivalent road octane number with respect to speed undoubtedly would have been quite different.

Furthermore, some engines depreciate the rating of sensitive fuels like cracked gasolines or diisobutylene-n-heptane reference fuels while others appreciate this type of fuel. Engines of the first type are called "severe"; the latter are called "mild". Engine severity is affected by a number of variables, the most important of which are summarized in Table 5.

TABLE 5

Engine Variable	Engine Severity Greatest when Variable is
Speed	High
Mixture Temperature	High
Jacket Temperature	High
Volumetric Efficiency	Low
Mixture Ratio	Lean
Distribution	Poor

It is important that the petroleum technologists studying the inherent antiknock characteristics of fuels on the road have some means of determining the severity of test engines. Now that the industry is committed to using the primary reference fuels, isooctane and n-heptane, in place of the old secondary reference fuels which are no longer available, it has been suggested that diisobutylene or some similar sensitive pure hydrocarbon be added to the list. It would then be possible, as suggested by W. G. Lovell of General Motors, to evaluate the degree of severity of the engines by determining the equivalent percentages of isooctane and diisobutylene in n-heptane which will give Borderline knock over the speed range. One problem in connection with the suggestion is that the diisobutylene blends would not be of constant sensitivity, and it is readily apparent that this factor may be significant.

It is not the purpose here to enter into a thorough discussion of all the factors which influence the fuel-engine relation. This would take as much time as already has been devoted to this presentation. It is important only that the petroleum industry recognize the fact that fuel performance is inseparable from engine performance and test method.

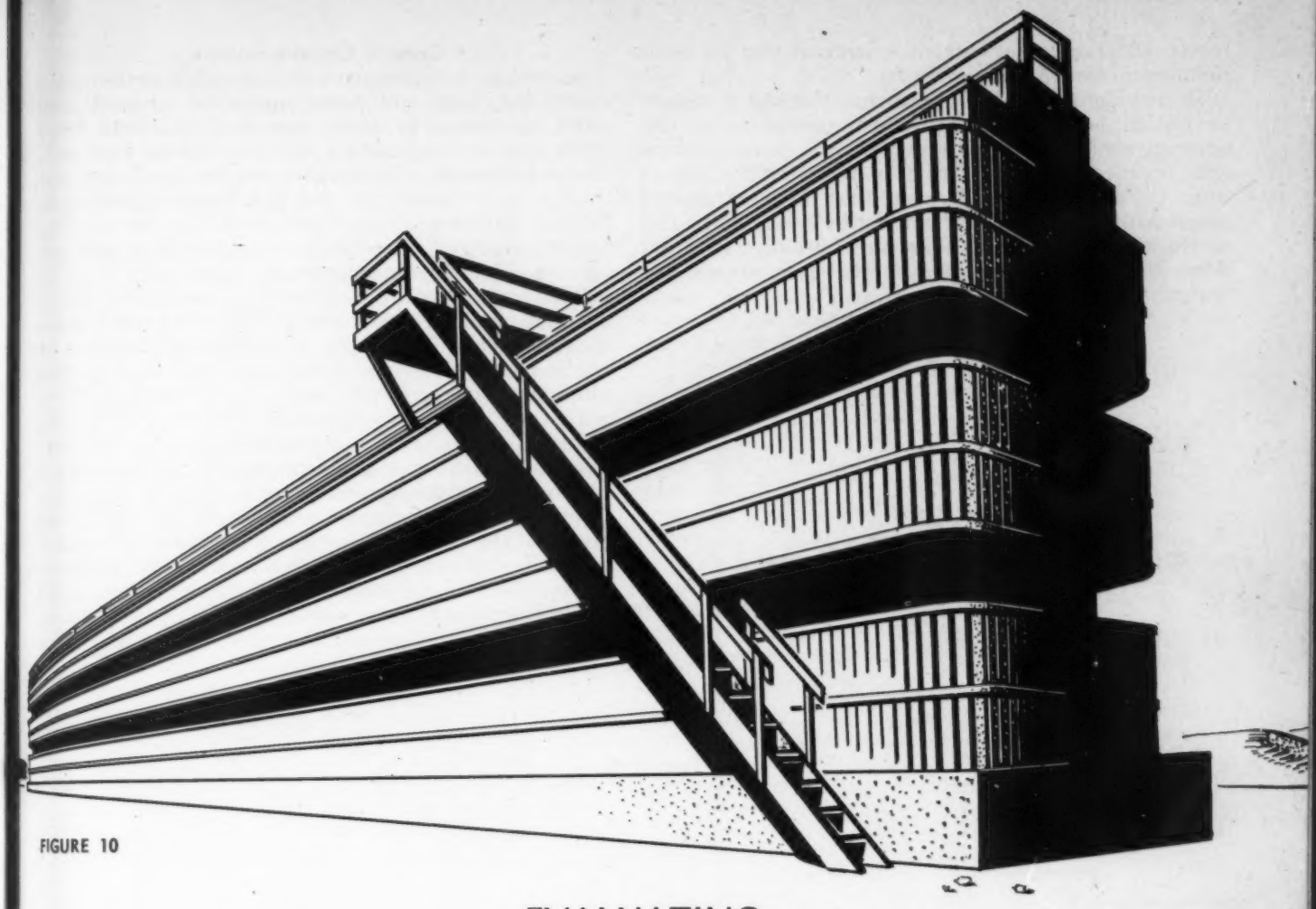


FIGURE 10

EVALUATING

Cooling Tower Performance

FOR THE PROCESS MAN

JAMES G. DeFLON
Chief Cooling Tower Development Engineer
The Fluor Corporation, Ltd.

THE processes of cooling water are among the oldest and simplest known to man. All that is required to cool water is to expose its surface to air. Some of these cooling processes are slow, such as the cooling of water on the surface of a pond; while others are comparatively fast, such as the spraying of water into air. These processes all involve the exposure of water surface to air with varying degrees of efficiency.

The heat transfer process involves a latent heat transfer due to change of state of a small portion of the water from liquid to vapor and a sensible heat transfer due to the difference in temperature of water and air. Approximately a thousand Btu are required to evaporate one pound of water, which is the amount of heat lost in cooling 100 pounds of water 10°. Therefore, for each 10° of cooling effected, roughly 1 percent of water is lost by evaporation. In addition, there will be a spray loss of not more than 0.2 percent in a well-designed atmospheric or mechanical-draft tower. Water cooling from 120° to 90°, for example, will lose 3.2 percent of its weight (3 plus .2 percent) with each passage through the tower.

In cooling towers in which the water is warmer than the air, the heat removed from the water and transferred to the air is the sum of the sensible heat and the latent heat of evaporation. The sensible heat q_s is small compared to the latent heat transferred, wr.

$$q_t = qs + wr$$
$$w = kA\Delta p_m$$

The factors which influence the performance of a cooling tower are therefore those which affect the expression $kA\Delta p_m$. The value of kA depends on the construction of the equipment, the extent of water surface exposed through drop surface and filming surface, and the velocity of the air. Δp_m depends on the temperature of the water and the temperature and humidity of the air. Actually Δp_m represents the difference in vapor pressure between the water at its

temperature and the water if it were at the wet-bulb temperature of the air.

It is evident from the above that the water cannot be cooled below the wet-bulb temperature of the entering air. The wet-bulb temperature, or to be more precise, the adiabatic saturation temperature, represents the minimum temperature that the water would reach with infinite time of contact between water and air in a cooling tower. This must be kept in mind when designing a plant to operate on cooling-tower water.

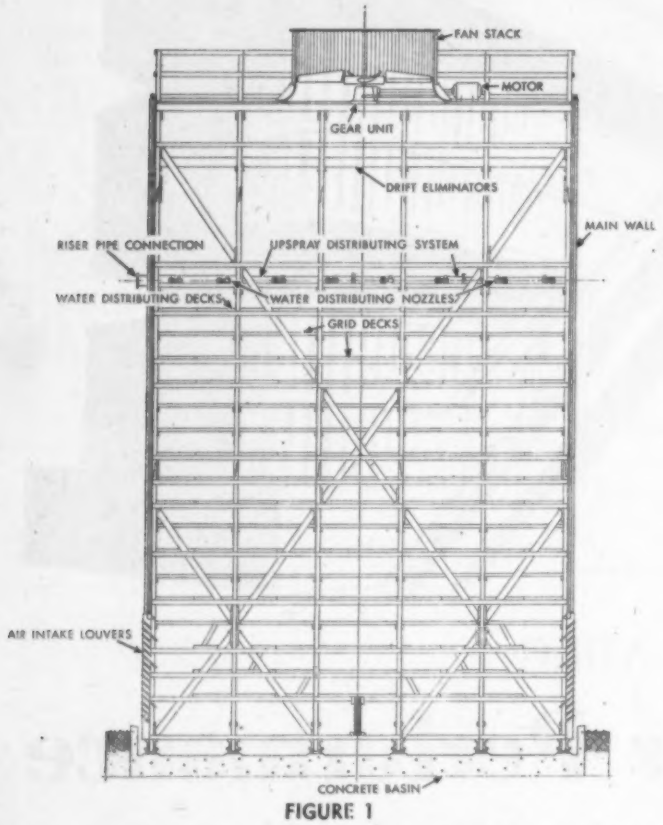


FIGURE 1

Cross-sectional view of a mechanical-draft cooling tower. With the upspray distributing system, it is necessary to allow sufficient space between the drift eliminators and the nozzles for a spray chamber.

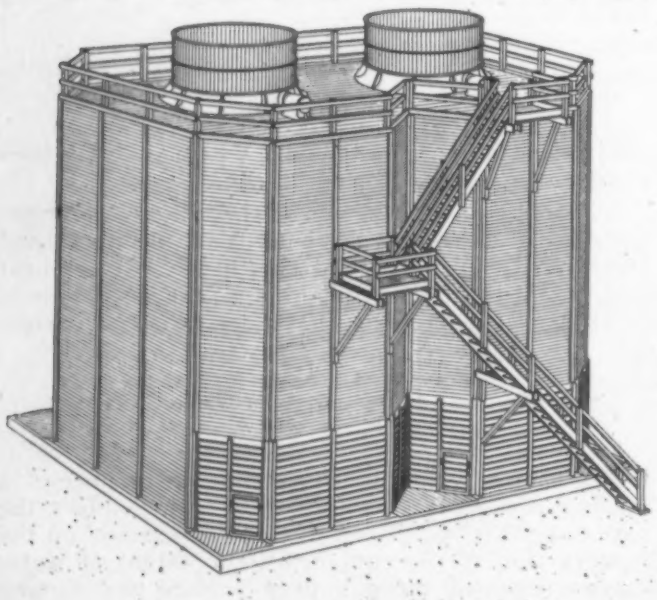


FIGURE 2

A modern two-cell mechanical-draft cooling tower. Note the square corners have been removed to facilitate air distribution within structure.

General Considerations

As process refinements have appeared, industry has demanded closer and closer control of its production units, resulting in more exacting demands being made upon cooling towers. In many plants, each additional degree of cooling has meant hundreds and even thousands of dollars per day in increased production. It has, therefore, been necessary for the cooling-tower industry to produce a tower which would supply the colder water more economically.

There are two types of cooling towers in general use today—the atmospheric (Figure 10) and the mechanical draft (Figure 2). The older apparatuses for cooling water, i.e., the spray pond and natural-draft chimney towers, have been almost exclusively replaced by these two types of cooling towers. The objection to the spray pond is the limited performance available and the nuisance created by the high water loss occurring during certain seasons of the year. The objection to the natural-draft tower is the high initial cost and the serious reduction in performance experienced during periods of hot weather. Both the atmospheric and the mechanical draft towers are capable of cooling water to the same minimum temperatures. The economic situation, the prevailing atmospheric

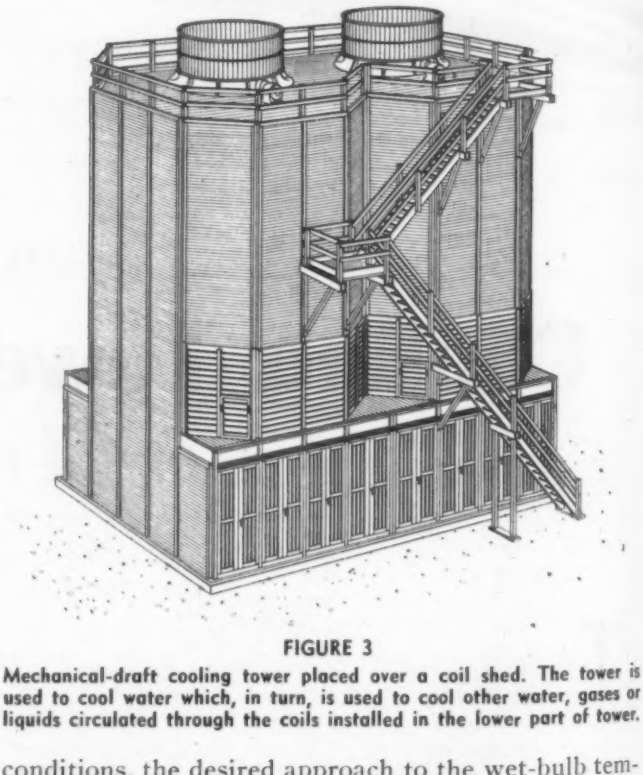


FIGURE 3

Mechanical-draft cooling tower placed over a coil shed. The tower is used to cool water which, in turn, is used to cool other water, gases or liquids circulated through the coils installed in the lower part of tower.

conditions, the desired approach to the wet-bulb temperature, and the amount of space available, will indicate which type to select.

Mechanical Draft Towers

There are two types of mechanical-draft towers in use today—the forced-draft and the induced-draft. In the forced-draft tower, the fan is mounted at its base and the air is forced in the bottom and discharged through the top at low velocity. In the induced-draft tower, the fan is mounted on the roof of the structure and air is pulled upward and discharged at a high velocity.

The forced-draft type is fast losing favor as it is more often subjected to the recirculation of the hot humid exhaust vapors back into the air intakes than the induced-draft type. This occurs under certain

atmospheric conditions because the velocity of the humid exhaust air is so low that the suction created by the fans tends to draw it back into the tower. Since the wet-bulb temperature of the exhaust air is considerably above that of the ambient air, there is a decrease in performance evidenced by an increase in cold-water temperature.

Except for the location of the fans, the structural and operational features of the two types of mechanical-draft towers are essentially the same. A cross-sectional view of the induced-draft tower with the various parts labeled is shown in Figure 1. The entrained moisture is removed from the exhaust air by the drift eliminator, which is placed just above the spray chamber and below the fan. The water is pumped to the main header located in the top of the tower where it is distributed to the various nozzles. This water is sprayed up in a manner similar to that used in a spray pond, and is intimately mixed with the exhaust air before dropping to the docks below. (In performance, the upspray distributing system represents the equivalent of adding 8 or 9 feet to the height of the cooling tower over that of the gravity-type system.) The fall of the water is interrupted by the slat-type grids as it flows counter-currently to the air. In flowing counter-currently, the coldest water contacts the driest air and the warmest water contacts the most humid air. Maximum performance is thus obtained, since the temperature of all of the cold water is approaching the wet-bulb temperature of the entering dry air. This was not true of the older cross-flow and parallel-flow type cooling towers.

The performance of a given type cooling tower is governed by the ratio of the weights of air to water and the time of contact between water and air. In commercial practice, the variation in the ratio of air to water is first obtained by keeping the air velocity constant at about 350 feet per minute per square foot of active tower area, and varying the water concentration (gallons per minute per square foot of tower area). As a secondary operation, the air velocity is varied to make the tower accommodate the cooling requirement. The time of contact between water and air is governed largely by the time required for the water to discharge from the nozzles and fall through the tower to the basin. The time of contact is, therefore, obtained in a given type of unit by varying the height of the tower. Should the time of contact be insufficient, no amount of increase in the ratio of air to water will produce the desired cooling. Therefore, it is necessary that a certain minimum height of cooling tower be maintained. Where a wide approach of 15° to 20° to the wet-bulb temperature and a 25° to 35° cooling range* is required, a relatively low cooling tower will suffice. A tower in which the water travels 15 to 20 feet from the distributing system to the basin is sufficient. Where a moderate approach of 8° to 15° and a cooling range of 25° to 35° is required, a tower in which the water travels 25 to 30 feet is adequate. Where a close approach of 4° to 8°, with a 25° to 35° cooling range is required, a tower in which the water travels from 35 to 40 feet is required. It usually is not economical to design a cooling tower with an approach of less than 4°, but it can be satisfactorily accomplished with a tower in which the water travels 35 to 40 feet.

Figure 4 shows the relationship of the hot-water, cold-water and wet-bulb temperatures to the water concentration. From this, the minimum area required for a given performance of a well-designed counter-flow induced-draft cooling tower can be obtained. Figure 5 gives the horsepower per square foot of tower area required for a given performance. These curves do not apply to parallel or cross-flow cooling since these processes are not as efficient as the counter-flow process. Also they do not apply where the approach to the cold-water temperature is less than 5°. These charts should be considered approximate and for preliminary estimates only. Many factors not shown in the graphs must be included in the computation and hence the manufacturer should be contacted for final design recommendations.

The cooling performance of any tower containing a given depth of filling varies with the water concentration. It has been found that the maximum contact and performance is obtained with a tower having a water concentration of 2 to 3 gallons of water per minute per square foot of ground area. Thus, the problem of calculating the size of a cooling tower becomes one of determining the proper concentration of water required to obtain the desired results. A higher tower will be required if the water concentration falls below 1.6 gallons per square foot. Should the water concentration exceed 3 gallons per square foot, a lower cooling tower may be used. Once the necessary water concentration is obtained, the tower area can be calculated by dividing the gallons per minute circulated by the water concentration gallons per square foot. The required tower size then is a function of the following:

1. Cooling range (hot-water temperature minus cold-water temperature.)
2. Approach to wet-bulb temperature (cold-water temperature minus wet-bulb temperature).
3. Quantity of water to be cooled.
4. Wet-bulb temperature.
5. Air velocity through the cell.
6. Tower height.

To illustrate the use of the charts let us assume that we have the following cooling conditions:

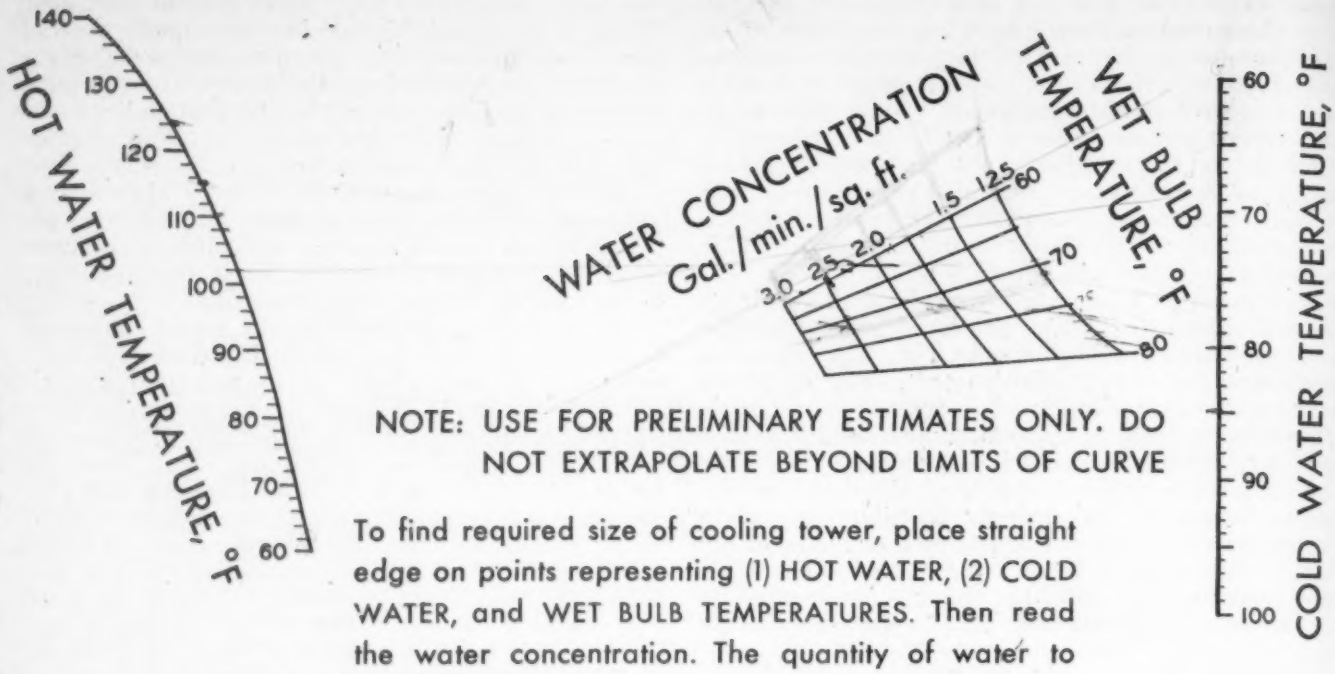
Hot-water temperature	= 102°
Cold-water temperature	= 78°
Wet-bulb temperature (Twb)	= 70°
GPM	= 2000

laying a straight-edge across Figure 4 and connecting the points representing the design water- and wet-bulb temperatures, we find that a water concentration of 2 gallons per square foot is required. Dividing the quantity of water circulated by the water concentration, we find that the theoretical area of the tower is 1000 square feet.

To obtain the theoretical fan horsepower, we use Figure 5. Connecting the points representing the 100 percent of standard tower performance with the turning point, we find that it will require .041 horsepower per square foot of actual effective tower area. Multiplying by the tower area of 1000 square feet, we find that it requires 41.0 fan horsepower to perform the necessary cooling.

Suppose that the commercial tower size is such that the actual tower area is 910 square feet. We can still obtain the cooling equivalent to 1000 square feet of standard tower area by increasing the air velocity through the tower. Within reasonable limits, the shortage of actual area can be compensated for by an increase in air velocity through the tower which, in turn, requires a higher fan horsepower. Our problem then becomes one of increasing the performance of the smaller tower by 10 percent. From Figure 5, by connecting the points representing 110 percent of

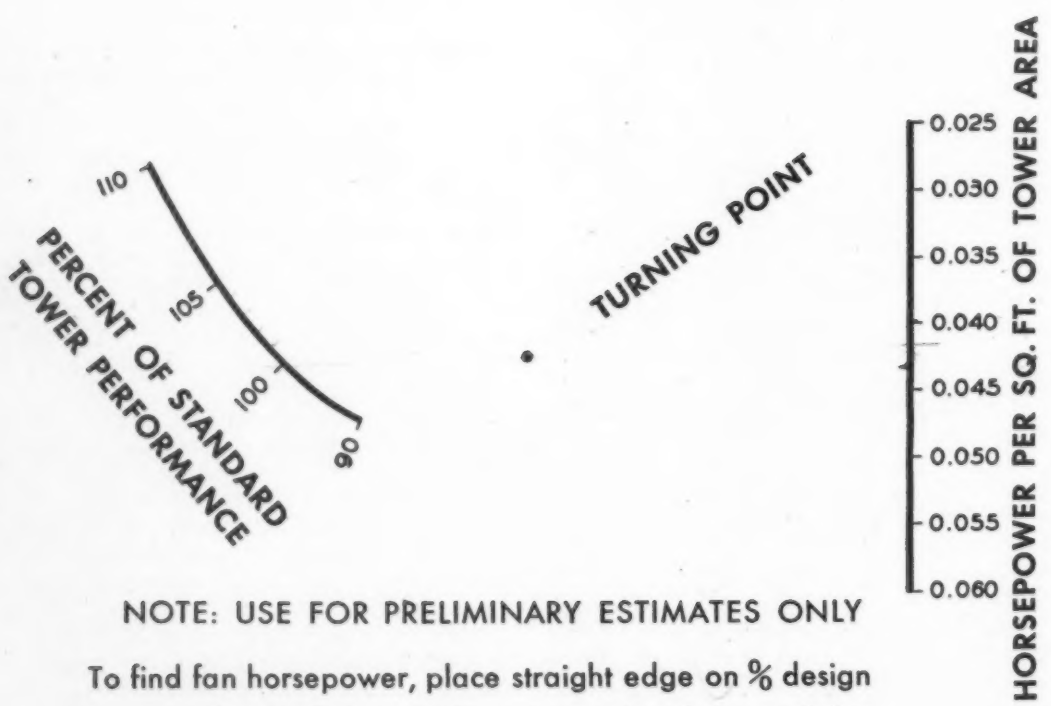
* The approach is the difference between the cold-water temperature and the wet-bulb temperature. The cooling range is the difference between the hot-water temperature and the cold-water temperature.



NOTE: USE FOR PRELIMINARY ESTIMATES ONLY. DO NOT EXTRAPOLATE BEYOND LIMITS OF CURVE

To find required size of cooling tower, place straight edge on points representing (1) HOT WATER, (2) COLD WATER, and WET BULB TEMPERATURES. Then read the water concentration. The quantity of water to be cooled divided by the water concentration, gives effective ground area of the cooling tower required.

FIGURE 4
Mechanical-draft cooling tower sizing chart.



NOTE: USE FOR PRELIMINARY ESTIMATES ONLY

To find fan horsepower, place straight edge on % design tower capacity factor and turning point, then read fan horsepower per square foot of tower area at right. Multiply tower area by this factor to obtain fan horsepower.

FIGURE 5
Mechanical-draft cooling tower chart.

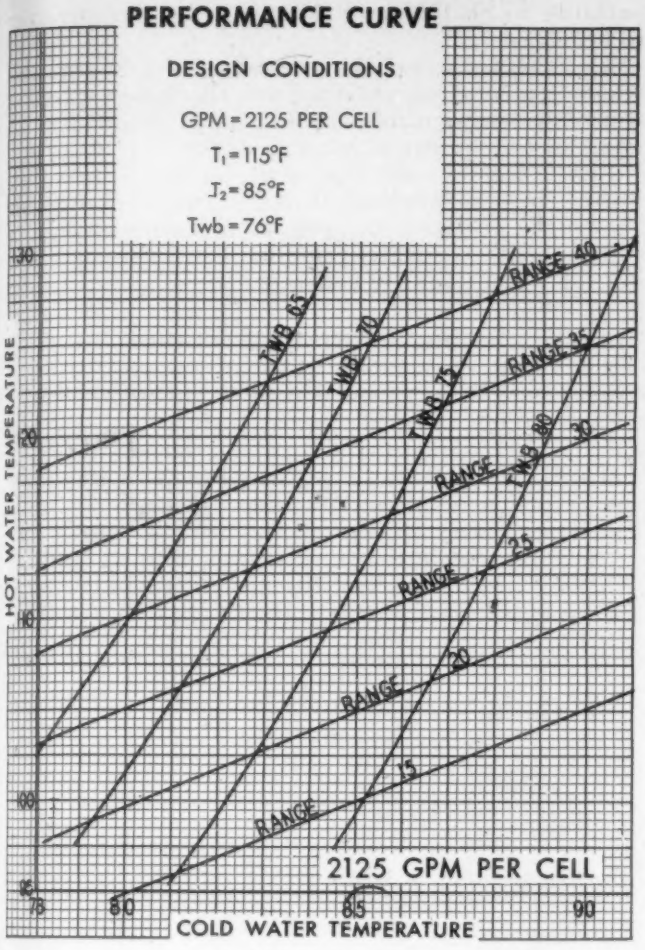


FIGURE 6

Typical performance curve for a mechanical-draft cooling tower.

standard tower performance and the turning point, the fan horsepower is found to be .057 horsepower per square foot of actual tower area, or .057 × 910 = 51.9 horsepower.

On the other hand, suppose the commercial tower size is such that the actual tower is 1110 square feet, the cooling equivalent to 1000 square feet of standard tower area can be accomplished with less air and less fan horsepower. By the use of Figure 5, the theoretical fan horsepower for a tower doing only 90 percent of standard performance is found to be .031 per square foot of actual tower area or 34.5 horsepower. This illustrates how sensitive the fan horsepower is to small changes in tower area. The importance of designing a tower which is slightly oversize in ground area becomes immediately apparent.

Assume the same cooling range and approach as used in the first example, except that the wet-bulb temperature is lower. The design conditions then would be:

GPM = 2000
Range = 24°
Approach = 8°
T_1 = 92°
T_2 = 68°
Wet-bulb temp.-(Twb) = 60°

From Figure 4, we find the water concentration required to perform the cooling is 1.75, giving a theoretical tower area of 1145 square feet as compared with 1000 square feet for a 70° wet-bulb temperature. This shows that the lower the wet-bulb temperature for the same cooling range and approach, the larger the area of the tower required, and therefore the more difficult the cooling job.

The problem of estimating the performance of an existing tower at other than design conditions often is encountered by the plant operator. For example, suppose we have a tower that was designed for the following conditions:

GPM = 1000
Range = 30°
Approach = 10°
T_1 = 110°
T_2 = 80°
Wet-bulb temp. = 70°

What will the cold water temperature (T_2) be when the wet bulb temperature (Twb) drops to 60°, provided of course, that the heat load and water quantity remain constant? From Figure 4, we find

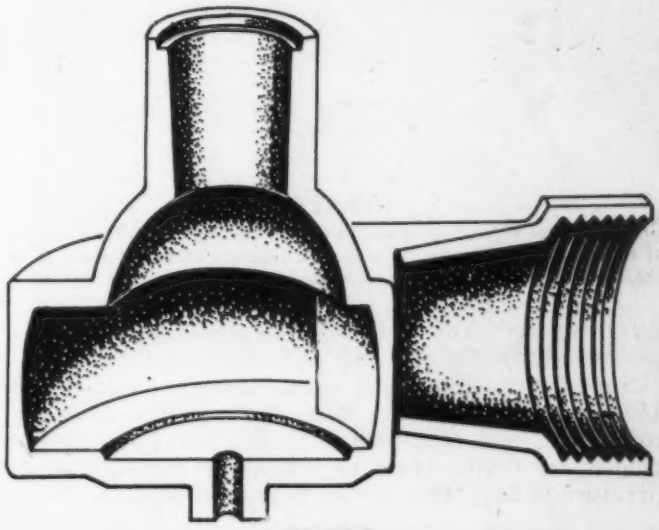


FIGURE 7

This cutaway view of an upspray nozzle shows the side entrance which starts the spiral action upward to the discharge orifice. The removable screw plate can be drilled as shown for automatic drainage to prevent winter freezing.

Nozzle Capacity Table

Nozzle No.	Pipe Size	Orifice Size	PRESSURE IN POUNDS PER SQUARE INCH 5	6	7	8	9	10	12	15	20	25	30
			CAPACITY IN U. S. GALLONS PER MINUTE										
2.4	⅜"	7/64"	....	....	1.0	1.1	1.2	1.3	1.4	1.6	1.8	2.0	2.2
2.4	⅜"	7/64"	1.8	1.9	2.1	2.2	2.3	2.3	2.7	3.0	3.4	3.8	4.2
7	1"	7/64"	6.5	7.1	7.7	8.2	8.7	9.2	10.1	11.3	13.1	14.6	16.0
14	1"	5/32"	11.5	12.6	13.6	14.5	15.4	16.2	17.7	19.8	22.7	25.3	27.8
19	1½"	3/16"	16.8	18.3	19.8	21.1	22.4	23.5	25.8	28.7	32.9	36.5	40.0
24	1½"	1"	20.0	22.0	23.8	25.3	26.9	28.3	31.0	34.7	40.0	44.8	49.0
39	2"	1¼"	33.0	36.2	39.0	41.8	44.3	46.7	51.0	57.0	65.8	73.3	80.0
49	2½"	1 11/32"	39.5	43.0	46.5	49.5	52.5	55.0	60.5	67.0	78.0	86.0	94.0

FIGURE 8

that the water concentration is 2.0 gallons per square foot at design conditions. This water concentration does not change since the volume of water and the tower area remain constant. With the water concentration at 2.0 and the wet-bulb temperature at 60°, by adjusting the angle of the straight edge on Figure 4 until we obtain a 30° differential between the hot-water and cold-water temperatures, we find the hot-water temperature to be 103°, and the cold-water temperature to be 73°.

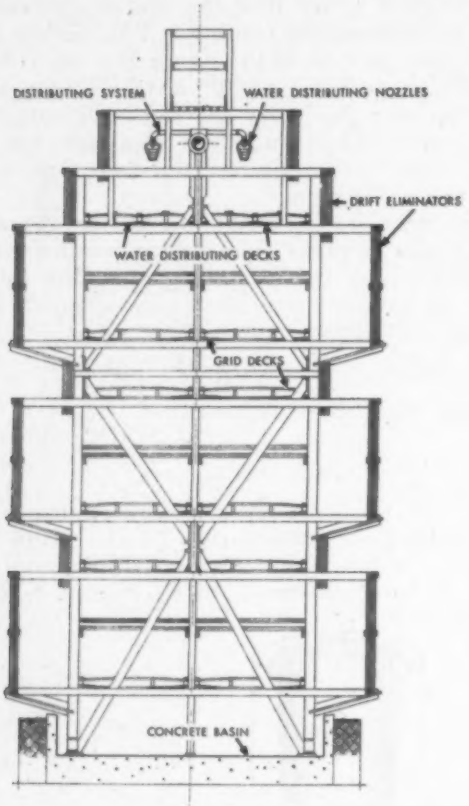


FIGURE 9

Cross-sectional view of an atmospheric-type cooling tower. The function of the drift eliminators on the side is to separate entrained water from the discharging air stream, reducing airborne drift loss to a minimum.

Suppose now, that the above designed tower had 1500 gallons per minute flowing through it, and the total heat load remained constant, what would the cold-water temperature be when the wet-bulb temperature is 65°? The design heat load was

$$1000 \times 8.33 \times 30 = 250{,}000 \text{ Btu per minute}$$

The new cooling range (heat load remaining constant) when circulating 1500 gallons per minute over the tower would be

$$\frac{250{,}000}{1500 \times 8.33} = 20.0°$$

Theoretically, the design area of the tower from Figure 4 was 500 square feet (1000 GPM ÷ 2.0 gal. per sq. ft. = 500 sq. ft.). The water concentration when circulating 1500 GPM is

$$\frac{1500}{500} = 3.0 \text{ gal. per sq. ft.}$$

Now, referring to Figure 4, with a water concentration of 3.0 gallons per square foot and 65° wet-bulb temperature, adjust the straight-edge until a difference of 20° exists between the hot-water and cold-water temperatures. This shows the hot-water temperature to be 100° and the cold-water temperature to be 80°.

This indicates that the possibility of a lower cold-water temperature obtained by the lower existing wet-bulb temperature was lost due to the adverse effect of the increased water quantity.

Figure 6 shows the type of performance curve furnished by the cooling tower manufacturer. This shows the variation in performance with change in wet-bulb and hot-water temperature, while maintaining the water quantity constant.

Cooling Tower Spray Nozzles

The type of nozzles used in up-spray cooling tower distributing systems is shown in Figure 7. The accompanying table in Figure 8 gives the performance of a particular family of nozzles. In up-spray distributing systems, a pressure of 7 pounds per square inch is common practice; however, 5 pounds is adequate.

This nozzle is of the non-clogging type. It does not depend on small orifices to obtain minimum drop-size but, rather, upon centrifugal force. The water is given a spiral action by its tangential entrance into the spiral chamber. The dome-shaped approach to the discharge orifice increases this spiral action as it approaches the discharge orifice. This whirling action, which generates the necessary velocity for fine break-up, assures uniform drop-size and efficient water distribution over a maximum area. This formation of uniformly small drops assures maximum contact with air, resulting in high cooling efficiency.

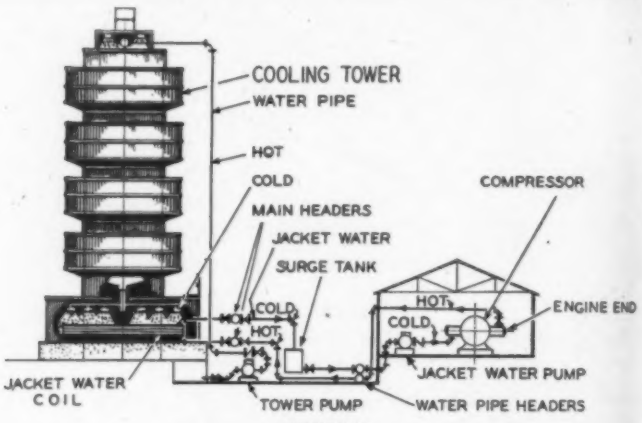


FIGURE 11

Typical flow diagram for a closed cooling system utilizing the coil-type atmospheric cooling tower.

Atmospheric Cooling Towers

A cross-sectional view of a typical atmospheric cooling tower is shown in Figure 9. In atmospheric towers, the water is pumped to the top of the tower, where it is discharged through a distributing system. As the water begins its downward flow, it is broken up and redistributed by the decks that comprise the filling of the tower. This continually creates newly exposed cooling surface for the air (passing horizontally through the tower) to encounter. The redistribution insures even concentration of water throughout the tower during its entire fall.

Although initial cost for an atmospheric cooling tower (designed for a three-mile wind) is about the same as that for a mechanical draft tower, there are certain important limitations governing its performance. It must be located broadside to the prevailing wind in an exposed area. Any surrounding structures,

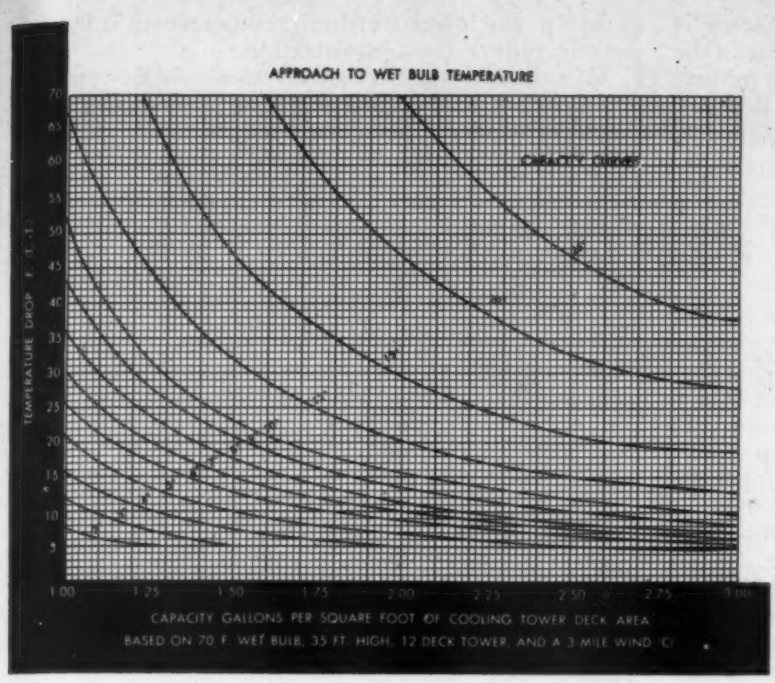

APPROACH TO WET BULB TEMPERATURE
CAPACITY CURVES
TEMPERATURE DROP °F
CAPACITY GALLONS PER SQUARE FOOT OF COOLING TOWER DECK AREA
BASED ON 70 F. WET BULB, 35 FT. HIGH, 12 DECK TOWER, AND A 3 MILE WIND

FIGURE 12
Capacity curves for atmospheric-type cooling towers.

hills or other barriers would tend to block off the wind.

Originally, the main objection to atmospheric towers was the excessive spray loss occurring during periods of high winds. This high loss was caused by the lack of a method of separating the entrained water from the air in the conventional louver-type tower. As a result, most manufacturers now have solved this problem by means of incorporating drift eliminators into the louvers. A typical example of this type of tower is shown in Figure 10.

The method of determining the size and performance of an atmospheric type tower is shown on the accompanying charts. The purpose of these charts is to show the method of estimating the approximate size of an atmospheric type cooling tower to meet a given demand. *The cooling capacity of any tower, with a given wet-bulb temperature and wind velocity, varies with the water concentration.* Thus, the problem of calculating tower size becomes nothing more than obtaining the correct water concentration for one of chosen height, which will operate under a certain wind velocity and wet-bulb

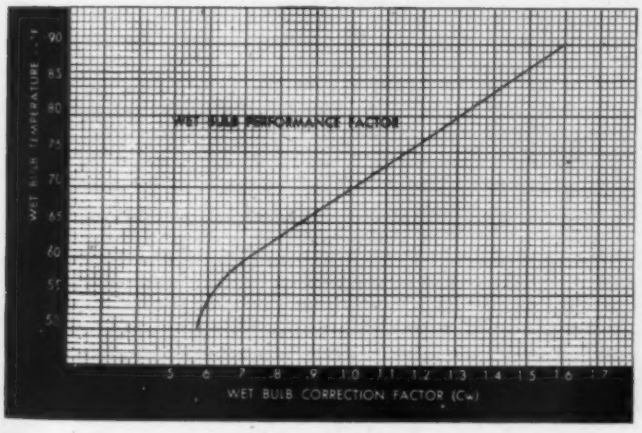


FIGURE 13
Wet bulb performance factor.

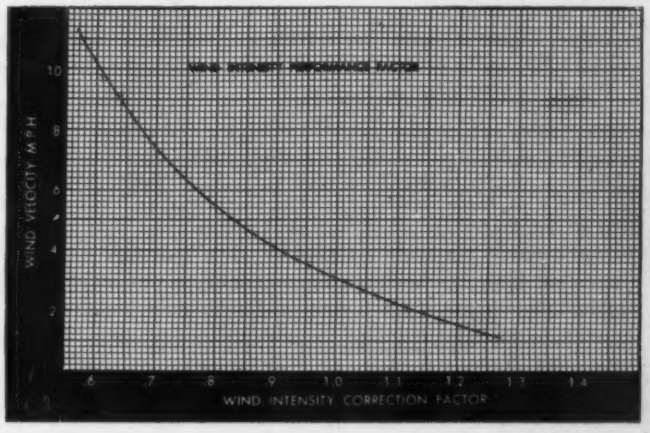


FIGURE 15
Wind intensity performance factor.

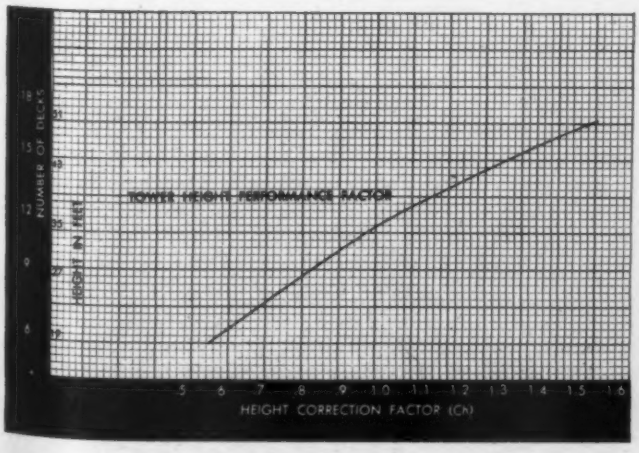


FIGURE 14
Tower height for performance factor.

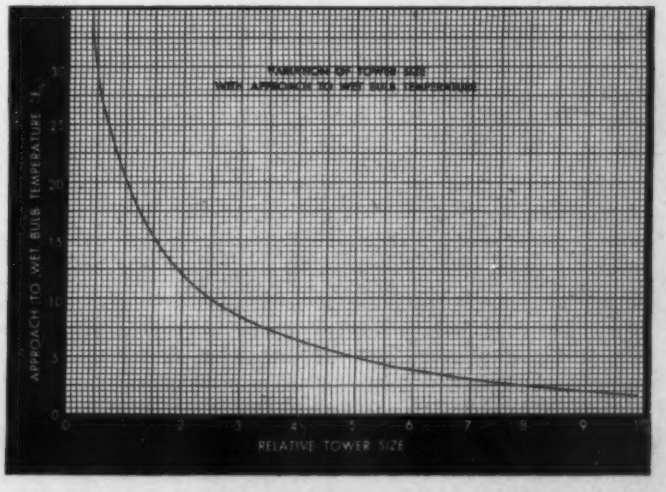


FIGURE 16
Variation of tower size with approach to wet-blub temperature.

temperature. Once this water-concentration factor is obtained, the area of a given height tower can be calculated easily by dividing the gallons per minute circulated by the concentration factor.

The concentration required to produce desired cooling depends primarily on the following conditions:

1. Temperature range ($T_1 - T_2$)
2. Approach to wet-bulb temperature ($T_2 - Twb$)
3. Tower height
4. Wind velocity
5. Wet-bulb temperature (Twb).

It is easily seen that because of the infinite number of possible combinations of these values, it is impractical if not impossible to have one curve from which to obtain the correct concentration factor. Figure 12 gives the required concentration for cooling water through a certain range and with a certain approach to the wet-bulb temperature. *but* this curve assumes a wet-bulb temperature of 70° F., tower height of 35 feet, and a wind velocity of 3 miles per hour. Let us assume that the conditions given are unity. Then should any of these three values change, the concentration would have to be corrected for the new conditions by using one or more of the correction factors shown on Figures 13, 14, and 15.

Let us see how a variance of any of the three aforementioned conditions would affect the concentration of water.

1. *Wind Velocity.* The higher the wind velocity the greater the amount of air that goes through the tower. This results in greater cooling. Therefore, when the wind velocity is higher, the concentration can be greater and still obtain equal cooling.*

2. *Tower Height.* In general, it is found in atmospheric as well as mechanical-draft towers that the greater the cooling range and the closer the approach to the wet-bulb temperature, the higher will be the tower required to give sufficient time of contact between water and air to accomplish the desired cooling. In atmospheric towers, the performance is limited by both maximum and minimum water concentrations. Should the water concentration fall below 1 gallon per minute per square foot of tower area, it will be necessary to employ the next size higher tower. This is because in extremely light water concentrations the water will not be uniformly distributed and the performance as predicted by the accompanying curves will not be achieved. Should the water concentration exceed 3 gallons per minute per square foot of tower area, it will then be necessary to choose the next size lower tower. The higher water concentrations tend to blanket the tower and will not allow sufficient air to pass through to accomplish the cooling shown on the curves.

3. *Wet-Bulb Temperature.* Theoretically, a cooling tower cannot cool water to a temperature lower than the prevailing wet-bulb temperature. Being limited by this fact, one is more interested in the economic approach of the cold-water temperature to said temperature. Air has a greater capacity for absorbing heat at the higher wet-bulb temperatures. At the lower wet-bulb, air, in passing through the tower, must have a greater temperature rise to accomplish the same cooling. Therefore, to obtain the same approach at the lower wet-bulb temperatures it is necessary to reduce the concentration.

To calculate the size of an atmospheric-type cooling tower with effective width of 12 feet, the following general formula may be used:

$$L = \frac{GPM \times W}{C \times 12 \times Cw \times Ch}$$

when:

L = Length of tower in feet.
GPM = Quantity of water in gallons per minute.
W = Wind correction factor.
C = Concentration of water per square foot of cooling-tower area.
Cw = Wet-bulb correction factor.
Ch = Tower-height correction factor.
T_1 = Inlet temperature.
T_2 = Outlet temperature.
($T_1 - T_2$) = Temperature range.
Twb = Wet-bulb temperature.
($T_2 - Twb$) = Approach to wet-bulb temperature.

Problem No. 1: Determine the length of a 35-foot-high tower required to cool 1500 gallons per minute from 90° F. to 75° F. with 70° F. wet-bulb temperature and a 3-mile-per-hour wind. From these conditions we know that the approach ($T_2 - T_w$) = 5° F. and the range ($T_1 - T_2$) = 15° F. From Figure 12, it is found that these two values require a concentration of C = 1.17. The correction factors, Ch, Cw and W will be equal to one, as shown on their respectipe correction factor curves, because Figure 12 was based upon values equal to those given in the problem. Substituting the corresponding values on the above formula we find that a tower 107 feet long is required.

Problem No. 2: Let us take the same conditions that exist in Problem No. 1 and see what the effect would be when the wind velocity is increased to 5 miles per hour. From Figure 15, when the wind velocity is 5 miles per hour, W = .80. Substituting this value for W in the formula, we find that an 85.3-feet-long tower is required.

Problem No. 3: Again assume the same conditions as Problem No. 1, except that due to space consideration it is necessary to have a 51-foot-high tower. From Figure 14, when tower height is 51 feet, Ch = 1.63. Substituting the new Ch in the formula we find that a 65½-foot-long tower is required.

Problem No. 4: Assume the conditions of this problem are identical to those of Problem No. 1, except that water has to be cooled from 85° F. to 70° F. and the wet-bulb temperature is 65° F. From Figure 13, when the wet-bulb temperature is 65° F., Cw = .86. Substituting this factor in the general formula we find tower length to be 124 feet.

Problem No. 5: Once a tower is installed the problem often arises of determining what cold-water temperature one can expect under conditions differing from those for which the tower was designed. For example, let us take the tower calculated for Problem No. 1. This tower is 35 feet high, 12 feet wide, and 107 feet long. What cold-water temperature, T_2, can be expected when the wet-bulb temperature is 60° F., wind velocity 4 miles per hour, cooling range 15° F., and water circulation 2000 gallons per minute?

The wet-bulb-temperature correction factor Cw from Figure 13 is .71. The wind-velocity correction factor (W) from Figure 15 is .875. By substituting these values in the general formula and solving for water concentration, we find:

$$C = \frac{2000 \times .875}{107 \times 12 \times .71 \times 1} = 1.92 \text{ gal. per sq. ft.}$$

Turning to Figure 12, we find that when the cooling range is 15° F. and the water concentration is 1.9, the approach to the wet-bulb temperature is 9° F. This means that the cold-water temperature will be 60° F. + 9° F. = 69° F.

The general information given is presented solely for the purpose of showing the more important factors that affect the design of atmospheric type cooling towers. Space does not allow consideration of the effect of excessive or insufficient concentration, higher relative humidity, change in wind direction, and other finer points which must be analyzed for proper cooling-tower selection. Therefore, it is to be noted that the cooling-tower sizes calculated in the above examples are only approximate. This survey, while presenting the essentials, has purposely been kept as non-technical as possible in order that it may serve as a guide to the plant operator in evaluating cooling-tower performance.

* In deciding upon a design wind condition, it is well to bear in mind that there is a period occurring during the day and night in which the prevailing wind shifts. When this occurs, there is a short period when there is little or no air movement. During such period, the tower-water temperatures will rise from 2 to 5 degrees, depending on the duration of the calm and the design wind velocity. This should be evaluated in determining a design wind velocity. However, one should be aware of the fact that many atmospheric towers are designed to successfully operate at a zero wind condition.

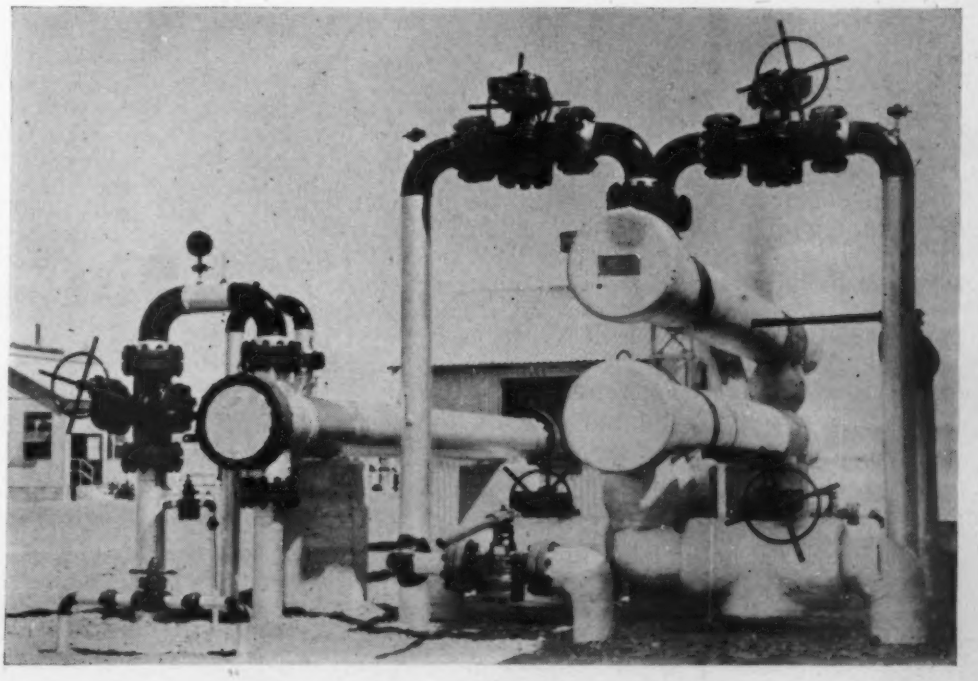
Refrigeration Removes Liquid Hydrocarbons from Gas In Transit

J. T. SIMON

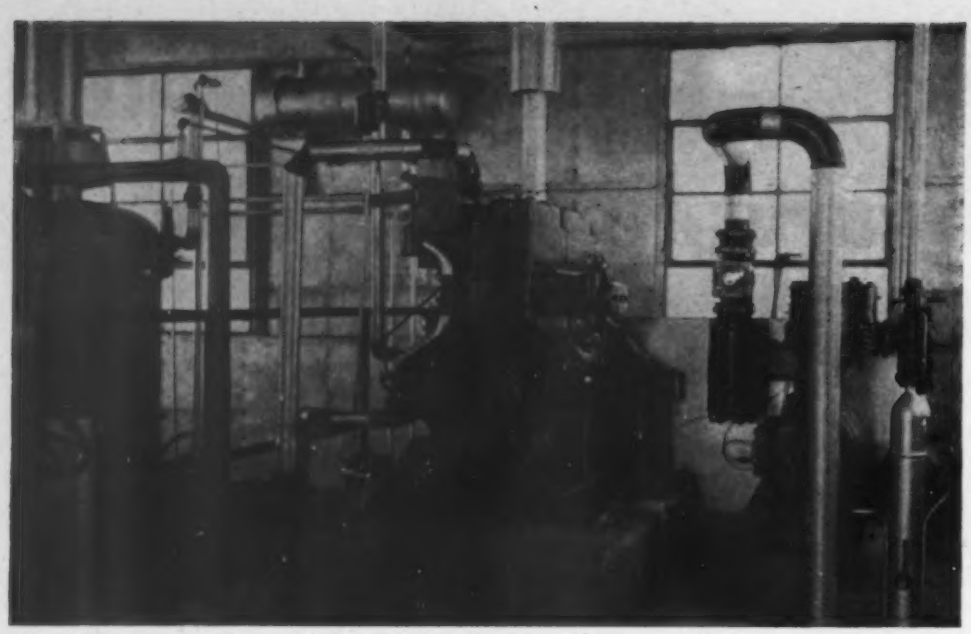
THE necessity for adequate dehydration of natural gas is well known in localities where pressure and temperature conditions are conducive to the formation of hydrates in natural-gas-transmission lines. Another problem, prevalent in some transmission lines, is the accumulation of hydrocarbon liquids at points of low line elevation, and the resulting decrease in line capacity accompanied by high operating pressures. This liquid accumulation can be eliminated by several methods; each has its advantages and disadvantages, and the particular method used must be the one that is best adapted to specific local conditions.

Much difficulty was experienced in operating an 8-inch transmission line, approximately 17 miles long, from the Powder Wash field, northwestern Colorado, operated by Mountain Fuel Supply Company. Hydrate formation caused some trouble, but hydrocarbon-liquid accumulation in the pipe line required periodic shutting-in of the field, in order to allow the liquid to flow by gravity to the lower points in the line where it was manually removed. Installation of more drips at low points on the line would have caught much of the fluid. However, some of the hydrocarbons were still in the gaseous state when they passed the last drip and would not condense out because the gas had not reached the minimum ground temperature. A properly designed scrubber, or drip, placed at a point on the pipe line where the gas had reached the lowest ground temperature, would have removed these liquids from the line. This drip apparently would have to be located several miles from the field, and several intermedate drips would be required to catch the liquid which condensed progressively as the flowing gas temperature approached ground temperature. One drip, or scrubber, was not sufficient, as the liquid which accumulated ahead of this drip caused an excessive pressure drop. Draining of these drips would require almost the full time of one man and a truck. The man-hours required, as well as the danger of fire, eliminated this method of solving the problem. Construction of a conventional gasoline plant for the processing of the gas from the field before entering the transmission line was not feasible from the standpoint of gas reserves and relatively low gasoline content of the gas.

Inasmuch as dehydration facilities were required to eliminate hydrates in the transmission line it was decided to install refrigeration equipment also to chill the gas to a temperature below the minimum



Gas-to-gas heat exchangers on right with refrigerant condenser on left.



Freon compressor at right, compressor engine left, and steam boiler for dehydration at left.

ground temperature. Chilling would condense the liquid hydrocarbons for removal from the gas before entering the main line. Gas compression equipment and electrical generators also installed in this plant do not come within the scope of this report and are not discussed.

The plant is designed to operate at a maximum pressure of 750 psig., and to handle 15,000,000 cubic feet of gas per day. With an inlet-gas temperature of 65° F., the plant will give a water dew point depression of more than 60° F., and will cool this volume of gas to 20° F. for the purpose of condensing the hydrocarbon liquids.

Gas Dehydration Cycle

The gas dehydration portion of this plant is of the typical liquid type, using diethylene glycol as the absorbing medium. One feature not found in regular dehydration plants of this type is the installation of two contactors in series. Such an installation insures that a minumum dew point is maintained at all times before the gas is subjected to cooling, so as to eliminate the danger of freezing in the gas chiller.

Wet gas enters the No. 1 contactor at the base, flows counter-current to the diethylene glycol, and discharges at the top of the contactor as dry gas. The gas then flows through a gas-to-gas shell and tube exchanger, and into the base of the No. 2 contactor. The two contactors are each 36-inch outside diameter by 24-foot, with a 13/16-inch shell and head thickness. Both have scrubber sections in the base and top; and any free liquid accumulating in the base is removed by steam traps. Warm gas from the No. 1 contactor is exchanged against the cold gas leaving the plant through the exchanger. Gas leaves the No. 2 contactor at an average temperature of 40° F. and a dew point of minus 10° F. The gas then enters a shell-and-tube chiller where the gas temperature is further reduced to 20° F., and additional liquid hydrocarbons condense. The cold gas and liquid hydracarbons then pass to a 30-inch by 8-foot by 11/16-inch shell gas scrubber, where the liquid is separated and removed from the gas stream to storage by means of a pilot-operated liquid-level controller. Gas leaves the top of the scrubber, and after passing through the gas-to-gas heat exchanger and refrigerant condenser, enters the pipe line with a low dew point, completely denuded of any liquid hydrocarbons that would condense out because of a low ground temperature.

Dry glycol is pumped onto the top bubble-cap tray in each absorber by a 1½-inch by 2-inch horizontal triplex pump driven by a 3-horsepower explosion-proof motor, with a V-belt drive. Identical circulation rates to each contactor are controlled by manually-operated throttling valves and direct reading rate-of-flow meters. The dry glycol, flowing counter-current to the wet gas, absorbs the moisture from the gas and the wet glycol collects on the chimney tray from which it leaves the contactor. The wet glycol leaves the No. 1 contactor at 60° F. and the No. 2 contactor at 39° F. The two streams are mixed and enter the glycol exchanger where the cold, wet glycol is exchanged against the hot, dry glycol from the still. After this heat exchange, the wet glycol enters the glycol still at 247° F. The glycol still is 18-inch OD by 30-foot by ¼-inch shell, with 15 bubble-cap trays, and operates at approximately 7 psig. A temperature of 320° F. is maintained at the base of the glycol still by means of a reboiler operating on steam from a 10-horsepower vertical boiler, which operates at 145 psig. A temperature controller maintains a constant temperature in the base of the still by controlling the fire in the boiler. Water vapor stripped from the wet glycol passes out of the top of the still and is discharged to the atmosphere through a pilot-operated back-pressure valve. Dry glycol leaves the base of the still at 295° F. and is exchanged against the wet glycol; it leaves the exchanger at 88° F. and enters the suction of the glycol circulating pump.

Gas Refrigeration Cycle

The refrigerating medium used in this plant is dichlorodifluoromethane (CCL_2F_2), more commonly known as Freon 12. This refrigerant was chosen rather than ammonia, as it is non-toxic, and non-inflammable; thus, it was possible to locate the refigerant compressor in the same building with the dehydration plant boiler.

The Freon compressor, rated at 381,700 Btu per hour at 660 rpm, has four cylinders, each having a 6-inch core and 5-inch stroke. The compressor is belt-driven by a 6-cylinder gas engine, which develops 160

brake-horsepower at 1400 rpm (sea-level rating). The hot gas from the compressor passes through a shell-and-tube refrigerant condenser, where the Freon gas is condensed by the cold natural gas leaving the plant. The liquid Freon flows into the top of the refrigerant surge tank, which serves as a storage tank for the Freon. From the base of this vessel the liquid flows through a small dehydrator, which removes any slight amount of moisture from the piping that might remain after initial evacuation of the system. Liquid Freon flows from the dehydrator through the refrigerant exchanger where the temperature drops by an exchange with cold Freon gas. The liquid Freon then flows into the base of the refrigerant flash tank through a throttling valve actuated by a liquid-level controller in the flash tank. The refrigerant flash tank is 24-inch outside diameter by 6-feet by ¼-inch shell, with a scrubber section in the top. The base of this vessel is directly connected to the base of the shell-and-tube gas chiller so that the controller maintains the same liquid level in each vessel. Natural gas flowing through the tubes of the chiller is cooled by the evaporation of the liquid Freon in the shell. Freon gas from the top of the chiller is combined with the Freon gas in the upper portion of the flash tank. After flowing through the scrubber section, the gas leaves the top of the flash tank at 9° F., passes through the refrigerant exchanger and thence to the suction of the Freon compressor.

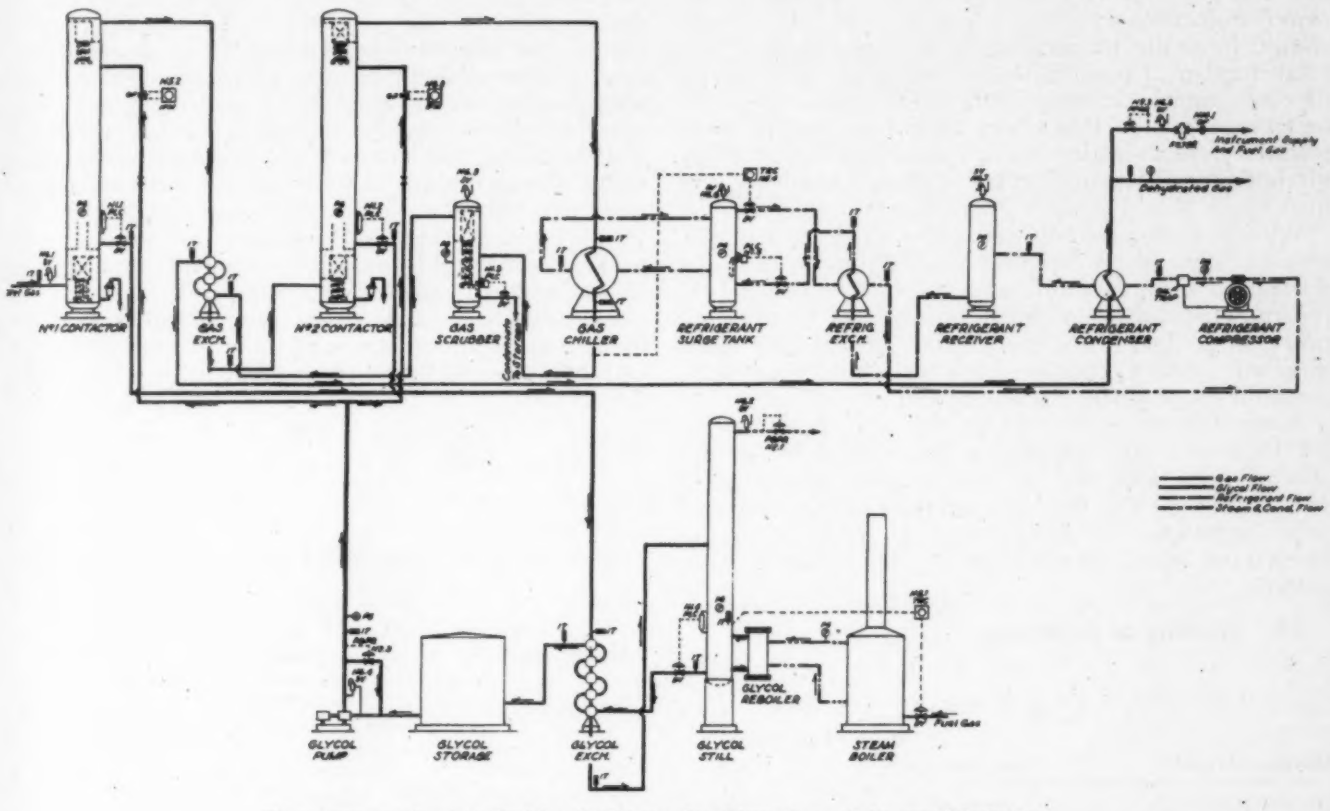
Control System

The natural gas leaving the chiller is maintained at a constant temperature by a temperature-record controller, which actuates a special packless-type motor valve. Should the natural gas when leaving the chiller drop below 20° F., this valve opens and allows hot Freon gas from the compressor discharge to by-pass directly to the gas chiller and maintain the specified temperature.

The boiler, glycol still and reboiler, glycol exchanger, and steam lines are insulated with magnesium pipe covering and blocks. The No. 2 contactor, gas chiller, gas scrubber, refrigerant flash tank, and connecting lines are insulated with 1½-inch cork and canvas.

Buildings

Because of extreme weather conditions in the Rocky Mountain area, it is necessary to house most of the equipment in a plant of this type. All equipment, except the gas exchanger and refrigerant condenser for the dehydration and refrigeration cycles, is housed in two buildings. Most of the equipment is located in the process building which is of steel-frame construction 24 feet by 36 feet by 12 feet, with corrugated iron roof and sides, lined with 28 gauge flat galvanized iron over ½-inch Celotex. The refrigerant compressor and steam boiler, together with the electric generators and jacket-water pumps for the natural-gas compressors are located in the auxiliary building. The size and construction of the auxiliary building are the same as the process building. These buildings are so constructed that the auxiliary building is kept warm by heat given off by the boiler and gas engines, and adequate heating of the process building is assured by leaving the insulation off one bank of the glycol exchangers.



Flow diagram, Mountain Fuel Supply Company's refrigeration unit for gas dehydration.

The plant has eliminated the formation of hydrates and condensation of liquid hydrocarbons in the main transmission line from the field in which the plant is located.

The author is indebted to W. T. Nightingale, vice president of Mountain Fuel Supply Company, for permission to publish this paper.

Chemicals From Petroleum

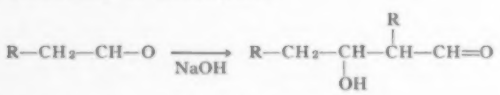
J. E. BLUDWORTH, Consulting Engineer

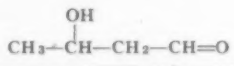
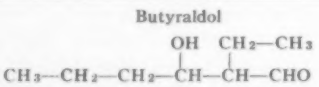
IT IS quite well known that acetaldehyde, acetone, methanol and formaldehyde can be obtained directly from natural gas. By other and as well known methods the higher alcohols, aldehydes and ketones also can be obtained from natural gas or some of its lower-molecular-weight components. If an attempt should be made to present, in any real degree, the total number of possibilities of chemicals from gas or its components, the magnitude of the endeavor would be tremendous. If this effort should be further complicated by elaboration on or differentiation between methods for the manufacture of these chemicals, the final result would be more confusing than informative.

We have taken, therefore, as the basis for our discussion some of the existing possibilities in the production of secondary products from a few of the better known primary or direct products of natural-gas processing. Too little space and effort have been directed toward consideration of the tremendous possibilities of aldehyde, ketone and alcohol uses.

Acetaldehyde is reacted in the operation labeled 3A to give aldol, which can be reacted in second operations labeled 1B, 2B, etc., to give other classes of compounds. The flow is continued until a series of four operations are shown here. In addition to the operations listed, product purification must be considered.

3A. Aldoling of aldehydes



Starting Material	Aldol Obtained	B.P.
Acetaldehyde	Acetaldol 	83° 20 mm
Butyraldehyde	Butyraldol 	High

This reaction will go for any aldehyde having an alpha hydrogen.

The aldols are very useful chemical intermediates. They can be used as a source of glycols, conjugated unsaturated aldehydes and ketones and beta hydroxy acids.

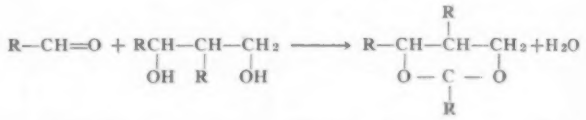
The aldols are formed in good yields. The aldols of aldehydes are sometimes difficult to separate from their corresponding aldehyde due to a cyclic acetal formation.

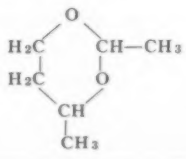
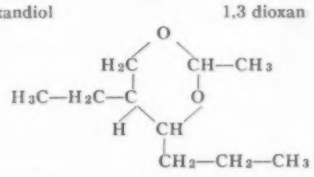
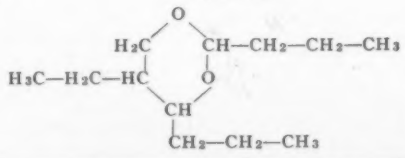
The formation of acetaldol from acetaldehyde is carried out in a slightly alkaline medium below room temperature. The medium is the reaction product, which is circulated through a reaction tank and a refrigeration exchanger to remove the heat of reaction. Acetaldehyde and small amounts of water and caustic are added to the recycle, and reaction product is withdrawn at the corresponding rate and acidified to stop the reaction.

The reaction rate is controlled by the temperature and alkalinity and is such that the reaction time is long enough that the reaction can be controlled. If the reaction is allowed to proceed too far, higher aldols than acetaldol are formed. Only about 60 percent of the acetaldehyde reacts to form acetaldol; the remainder forms a hemiacetal with acetaldol. The acetaldehyde in the hemiacetal cannot be removed from the reaction product by ordinary distillation, since the acetaldol dehydrates to crotonaldehyde under the influence of heat. By subjecting the mixture to vacuum at room temperature for an extended period the acetaldehyde can be stripped out, absorbed in water and recovered by distillation.

Acetals are formed from alcohols and aldehydes by heating in the presence of mineral acids and separating the acetal by fractionation after neutralizing the acid.

2A.



Starting Material	Product	B.P. °C.
Acetaldehyde and ethanol	Diethyl acetal $H_3C—CH<(OC_2H_5)_2$	103
Acetaldehyde and 1,3 butylene glycol	2,4 dimethyl, 1,3 dioxan 	150 est.
Acetaldehyde and 2 ethyl, 1,3 hexandiol	4,5 diethyl, 2 methyl 1,3 dioxan 	220 est.
Butyraldehyde and butanol	Dibutyl butryal $H_3C—CH_2—CH<(OC_4H_9)_2$	200
Butyraldehyde and 2 ethyl, 1,3 hexandiol	2,5 diethyl, 4 butyl 1,3 dioxan 	240 est.

WHEN this paper was presented before the South Texas Section of the American Institute of Chemical Engineers, Port Arthur, December 7, 1944, the author was director of research for Celanese Corporation of America. Since then he has resigned to engage in consulting work with headquarters in Corpus Christi.

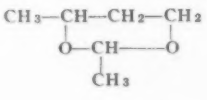
For several months it was under consideration for use in Chemical Engineering Progress. Decision to release the information through petroleum publications came in March and Petroleum Refiner took immediate advantage of the opportunity.

Diethyl acetal is formed by the reaction of acetaldehyde with ethyl alcohol in the presence of a trace of acid. The unreacted acetaldehyde and alcohol, after neutralizing the acid, are recovered by fractionation and recycled.

The acetals are liquids with ether-like properties, fairly stable to alkaline hydrolysis but unstable to acids. They can be used generally as solvents. The cyclic acetals, dioxolanes and dioxanes, have been used to prepare resins. The cyclic acetals are fairly stable and have solvent properties similar to dioxane.

The acetals are prepared easily and in good yields.

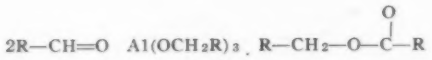
In a number of instances, products made from the starting materials in one or more operations could be reacted with the original reactants in a new series of operations. For instance, 1,3 butylene glycol, made through operations 3A and 2B, could be condensed with acetaldehyde by operation 2A to give 1,3 butylene glycol acetal.

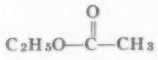
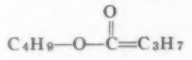
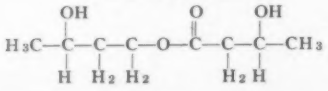


In such instances the number of operations required would be determined by studying the flow sheet.

The list of products is by no means complete, but it serves to illustrate the nature of the products obtained. Many other similar compounds not listed can be prepared, also, if desired.

6A. Tischenko condensation of aldehydes to esters



Aldehyde Used	Product	B.P.
Acetaldehyde	Ethyl acetate 	77
Butyraldehyde	Butyl butyrate 	166
Acetaldol	3 hydroxy butyl 2 hydroxy butyrate 	

This reaction is a general one for aldehydes but has not been commonly applied to manufacturing processes probably because of the difficulty in preparing catalysts.

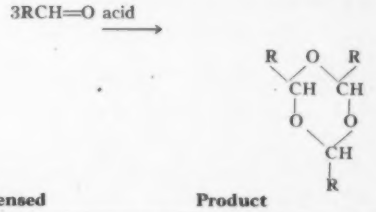
The reaction of two molecules of acetaldehyde to form ethyl acetate takes place in the liquid phase below room temperature in the presence of aluminum ethylate. Aluminum ethylate solution in ethyl acetate is prepared batchwise in a steel vessel equipped with a stirrer, reflux condenser and heating coils. A mixture of aluminum borings, aluminum chloride and ethyl acetate is heated to the boiling point, at which point the addition of absolute ethyl alcohol is started. Addition of the alcohol is continued over a period of 8-10 hours and reflux continued for 24 hours. The aluminum reacts completely to aluminum ethylate.

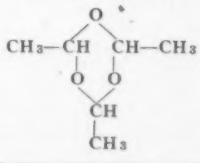
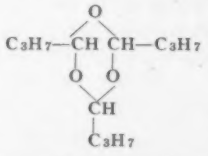
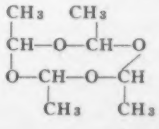
Equal weights of catalyst solution and anhydrous acetaldehyde are charged continuously to a brine-cooled reactor. The overflow from the first reactor goes to a second reactor maintained at a slightly higher temperature to complete the reaction. Since the catalyst is not recoverable, the reaction product is treated in an evaporator with enough water to hydrolyze it to aluminum hydroxide and ethanol. The evaporator separates the liquids from the aluminum hydroxide.

The purification system consists of four columns, the first of which fractionates off the unreacted acetaldehyde. The second column fractionates off the ternary ethanol-ethyl acetate-water azeotrope which is sent back to the evaporator where the water reacts with the unused catalyst. In the third column the overhead product is the binary azeotrope, ethanol-ethyl acetate, which is used for catalyst preparation. Finished ethyl acetate is the overhead product of the last column.

Mixtures of mixed esters also can be prepared by using a mixture of aldehydes as the reacting materials. For example, by condensing acetaldehyde and butyraldehyde together a mixture of esters would be formed containing ethyl acetate, butyl acetate, ethyl butyrate and butyl butyrate.

8A. Aldehyde condensations catalyzed by acids



Aldehyde Condensed	Product	B.P.
Acetaldehyde	Paraldehyde 	124°
Butyraldehyde	2,4,6 tripropyl 1,3,5 trioxan 	100° 35 mm
Acetaldehyde	Metaldahyde 	Dec. 100°

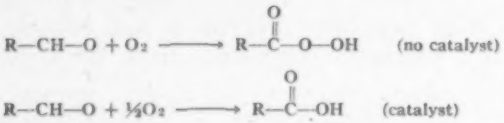
In acid media the simpler aldehydes are known to

go to their corresponding trimers in good yields. Acetaldehyde also will form a tetramer at low temperatures.

The compounds are unstable to acid or alkali and regenerate the aldehyde. For this reason they are often a good source of the anhydrous aldehyde and are easy to handle because of their lower volatility and stability. Trioxane is particularly useful as an anhydrous source of formaldehyde, because pure formaldehyde cannot be handled as a stable liquid.

Acetaldehyde is converted to paraldehyde by a trace of acid. The unreacted acetaldehyde can be fractionated off after neutralizing the acid.

9A. Oxidation of aldehydes



Aldehyde Oxidized	Product	B.P.
Butyraldehyde	Butyric acid	163°
Crotonaldehyde	Maleic anhydride	202°
Crotonaldehyde	Crotonic acid	185°
Acetaldol	B hydroxy butyric acid	dec.

Peracids made in the non-catalytic oxidation are difficult to separate from the aldehyde. They have been shown to be intermediates in the formation of the acids, and they must be handled very carefully to avoid explosions.

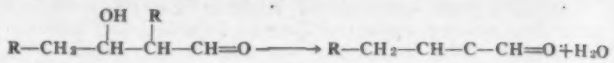
The unsaturated aldehydes have been oxidized to the corresponding acids, but the yields, in general, are not as good. Their conjugated double bonds make them useful intermediates for many chemical syntheses.

The aldols might also be oxidized under certain conditions to 3-hydroxy acids.

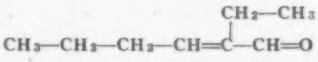
Maleic anhydride can be made in good yields by the vapor-phase oxidation of crotonaldehyde.

Acetaldehyde, as a dilute solution in acetic acid, is oxidized to acetic acid by air in the presence of a catalyst. Temperature control is obtained by recycling a large portion of the reaction product through water-cooled exchangers. The reactor product is fed into a column where unreacted acetaldehyde, water and low-boiling impurities are distilled overhead. The residue from this column then is fractionated to separate the pure product from catalyst and high boilers. The acetaldehyde vapor in the air from the reaction is removed by absorption in water. Acetaldehyde then is stripped from the aqueous solution in the usual manner.

1B. Dehydration of aldols



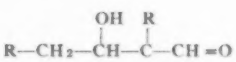
Starting Material	Product	B.P.
Acetaldol	Crotonaldehyde $CH_2—CH=CH—CH=O$	102°
Butyraldol	2 ethyl 3 propyl acrolein	180° est.



The aldols dehydrate easily and in good yields to give unsaturated aldehydes. The unsaturated aldehydes are useful intermediates and may be made to polymerize with peroxide catalysts. Since they can be made so easily, work on their polymerization may be worth investigation.

Acetaldol readily dehydrates to crotonaldehyde under the influence of heat in the presence of traces of iodine. Acetaldol containing a trace of iodine is fed into a column where acetaldehyde is distilled off. The residue goes to a second column where the crotonaldehyde-water azeotrope comes overhead and inorganic salts out the base. The azeotrope contains 80 percent by weight of crotonaldehyde.

2B. Hydrogenation of aldols



Starting Material	Products	B.P.
Acetaldol	1,3 butylene glycol	207°
Butyraldol	2 ethyl, 1,3 hydroxy hexane	270° est.

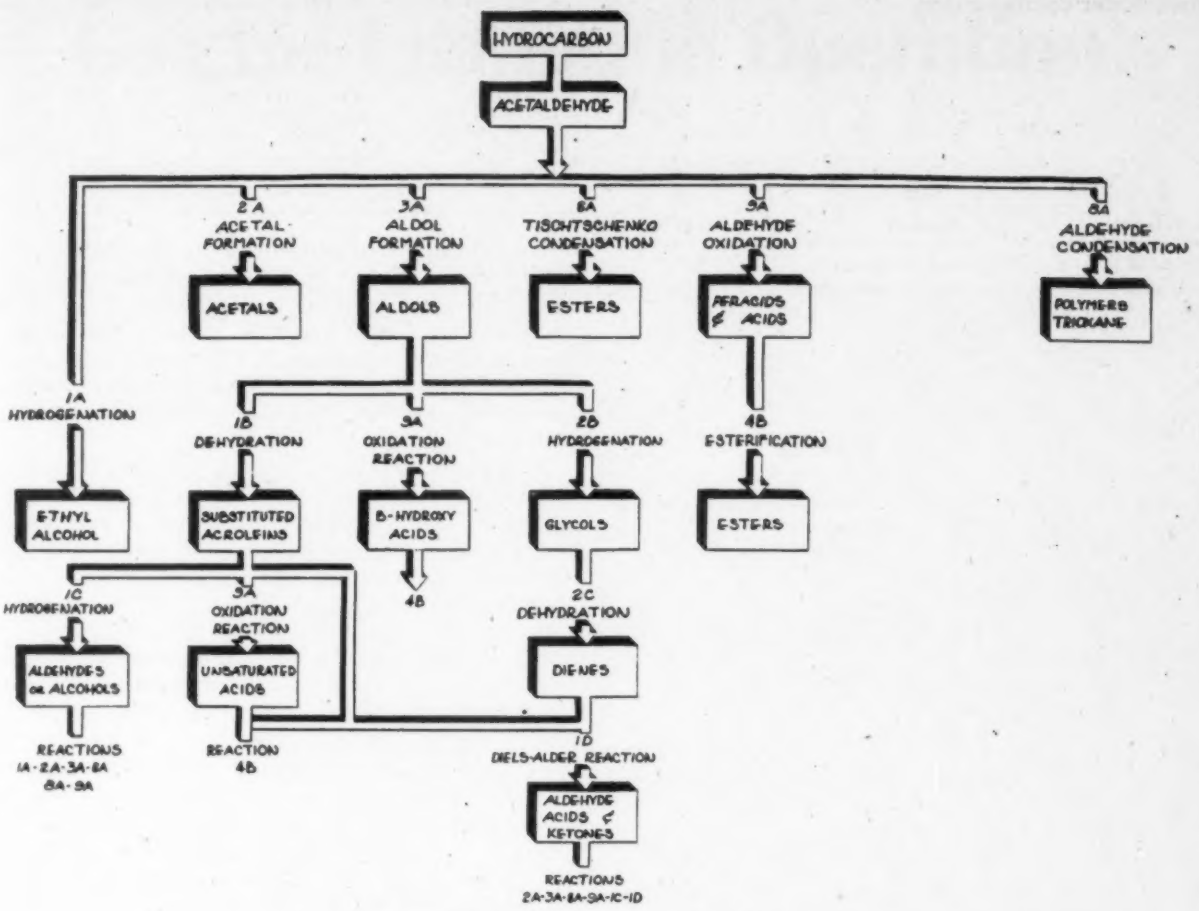
The aldols hydrogenate in good yields to glycols. The products are useful for making plasticizers, resins, explosives, solvents and as chemical intermediates.

Acetaldol is hydrogenated to 1,3 butylene glycol at 800 psi and temperatures ranging from 120° to 230° F. in the presence of nickel catalyst. The reaction is carried out continuously in a series of tall, small diameter reactors by passing acetaldol with catalyst in suspension cocurrently with a large excess of hydrogen. After cooling the product from the last reactor the excess hydrogen is separated and recycled to provide the agitation necessary to keep the catalyst suspended.

Each reactor is provided with a jacket through which mixtures of steam and water are passed to maintain the temperature of each reactor slightly higher than that of the preceding reactor. The temperature in the first reactor must be kept low because the high acetaldol concentration favors crotonization. A higher temperature is necessary in the last reactor to complete the hydrogenation.

Most of the catalyst fed to the reactors is that recovered from the hydrogenated product by settling and filtration. The make-up catalyst is freshly prepared by treating powdered nickel-aluminum alloy with caustic soda and then washing with water.

The hydrogenated product contains, in addition to 1,3 butylene glycol, ethanol from hydrogenation of acetaldehyde in the acetaldol feed and butyl alcohol from crotonization and hydrogenation of acetaldol. The purification system consists of an ethanol col-



Some of the derivatives from natural gas.

umn, butanol column and two glycol columns. The ethanol is fractionated from the hydrogenated product in the ethanol column. Butanol and water are then fractionated from the mixture in the butanol column. The residue from the butanol column is then distilled in the glycol columns under vacuum, leaving as a residue the higher boiling compounds formed as a result of side reactions in the aldoling process.

4B. Esterification

$$R{-}OH + R{-}\overset{O}{\overset{\|}{C}}{-}OH \longrightarrow R{-}O{-}\overset{O}{\overset{\|}{C}}{-}R + H_2O$$

Alcohol	Acid
1,3 butylene glycol 2 ethyl, 1,3 hexane diol butanol ethanol	acetic acid butyric acid crotonic acid 2 ethyl 3 propyl acrylic acid maleic acid tetra hydro phthalic acid

Ethyl acetate is produced by the reaction of acetic acid with ethanol in the presence of sulfuric acid which catalyzes the reaction. The reaction mixture is fed into a fractionating column from which the ternary azeotrope of ethanol-water-ethyl acetate comes overhead. This distillate is diluted with water which causes two layers to separate, the top being nearly pure ethyl acetate. The bottom which is dilute ethanol is separated in a second fractionation.

1C. Hydrogenation of substituted acroleins

$$H_2 + R{-}CH_2{-}CH{=}\overset{R}{\overset{|}{C}}{-}CHO \xrightarrow[\text{acid}]{} R{-}CH_2{-}CH_2{-}\overset{R}{\overset{|}{C}}H{-}CHO$$

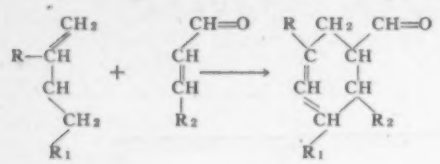
$$2H_2 + R{-}CH_2CH{=}\overset{R}{\overset{|}{C}}{-}CHO \xrightarrow[\text{neutral}]{} R{-}CH_2{-}CH_2{-}\overset{R}{\overset{|}{C}}H{-}CH_2OH$$

Starting Material	Product	B.P. °C.
Crotonaldehyde	Butyraldehyde $CH_3{-}CH_2{-}CH_2{-}CHO$	75.7
Crotonaldehyde	1 butanol $CH_3{-}CH_2{-}CH_2{-}CH_2{-}OH$	117.7
2 ethyl, 3 propyl acrolein	2 ethylhexanol $CH_3{-}CH_2{-}CH_2{-}CH_2{-}\underset{\displaystyle CH_2}{\underset{\displaystyle |}{CH}}{-}CH_2{-}OH$ $\underset{\displaystyle CH_3}{\underset{\displaystyle |}{}}$	183.5

By control of pH this reaction can be made to yield either the alcohol or the aldehyde. The products listed could be reused in a number of the reactions already listed. Butanol, for instance, could be used as a source

of higher alcohols which have such uses as flotation agents, anti-foaming agents and plasticizers.

1D. Diels Alder condensation



Starting Material	Product	B.P. °C.
1,3 butadiene and crotonaldehyde	1,2,3,6 tetrahydro 1 methyl benzaldehyde	79(10)
1,3 butadiene and crotonic acid	1,2,3,6 tetrahydro 2 methylbenzoicacid	
1,3 butadiene and maleic anhydride	△ 3 tetra hydro phthalic anhydride	
2 ethyl, 1,3 hexadiene and crotonic acid	1,2,3,6 tetrahydro, 2 methyl 3,5 diethyl benzoic acid	

This reaction is a general one and proceeds with the unsaturated acids, vinyl ketones and substituted acroleins as well. It involves the combination of two compounds, both having conjugated unsaturation, to give a singly unsaturated ring compound.

Products having a wide range of physical and chemical properties can be made. The products are cyclic aldehydes, acids and ketones and could be used over in a number of the earlier described operations.

Butadiene can be made to react with crotonaldehyde by passing a mixture of the two through a reactor at about 100° C. About 30 per cent of the reactants are converted to 5-methyl — Δ^3 — tetrahydrobenzaldehyde. The unreacted butadiene is condensed from the vapor phase by cooling and is then recycled. The unreacted crotonaldehyde is distilled from the product and recycled.

2C. Dehydration of 1,3 glycols

Starting Material	Product	B.P.
1,3 butylene glycol	Butadiene $CH_2=CH-CH=CH_2$	1°
2 ethyl, 1,3 hydroxy hexane	2 ethyl, 1,3 hexadiene $CH_3-CH_2-CH=CH-C(CH_2-CH_3)=CH_2$	100° est.

The formation of butadiene by the dehydration of 1,3 butylene glycol has been studied extensively. Other 1,3 glycols may be dehydrated in a similar manner. The dienes are used in the Diels Alder synthesis of cyclic compounds. Dienes form useful polymers and are useful chemical intermediates because of the ease with which they add with compounds having an active hydrogen.

One, three butylene glycol is dehydrated to 1,3 butadiene in the vapor phase at atmospheric pressure and about 500° F. in the presence of catalysts. The reaction absorbs heat. Glycol and recycled butadiene are fed into a gas-fired furnace where the glycol is vaporized and the butadiene and glycol vapors superheated to the reaction temperature. The reactors are fin-tube exchangers in which the heat absorbed by the reaction is furnished by hot inert gas passing through the fin tubes. Catalyst is deposited on the fins.

By so conducting the reaction, the temperature drop is minimized. This is desirable since the highest yield is obtained within a small temperature range. Too low a temperature causes a low reaction rate while the upper temperature is limited by the polymerization of butadiene to oil.

The vapors from the reaction are cooled to room temperature, which causes the unreacted glycol, butenol, water and oil to condense. Most of the butadiene is recycled to the reactor for purposes of dilution. The butadiene product finally is washed with water to remove traces of aldehydes and then liquified by compression. The butadiene then is fractionated at 100 psig from higher-boiling impurities always present in very small amounts.

The products condensed from the reaction vapors are fed into a vacuum column where oil, water and butenol are fractionated from the unreacted glycol, which then is fractionated under vaccum from high-boiling material. The mixture of oil, water and butenol is washed with water in a disc-and-ring extractor to recover the butenol from the oil. Butenol is fractionated from the aqueous solution and returned to the reactor since it is an intermediate product of the overall reaction.

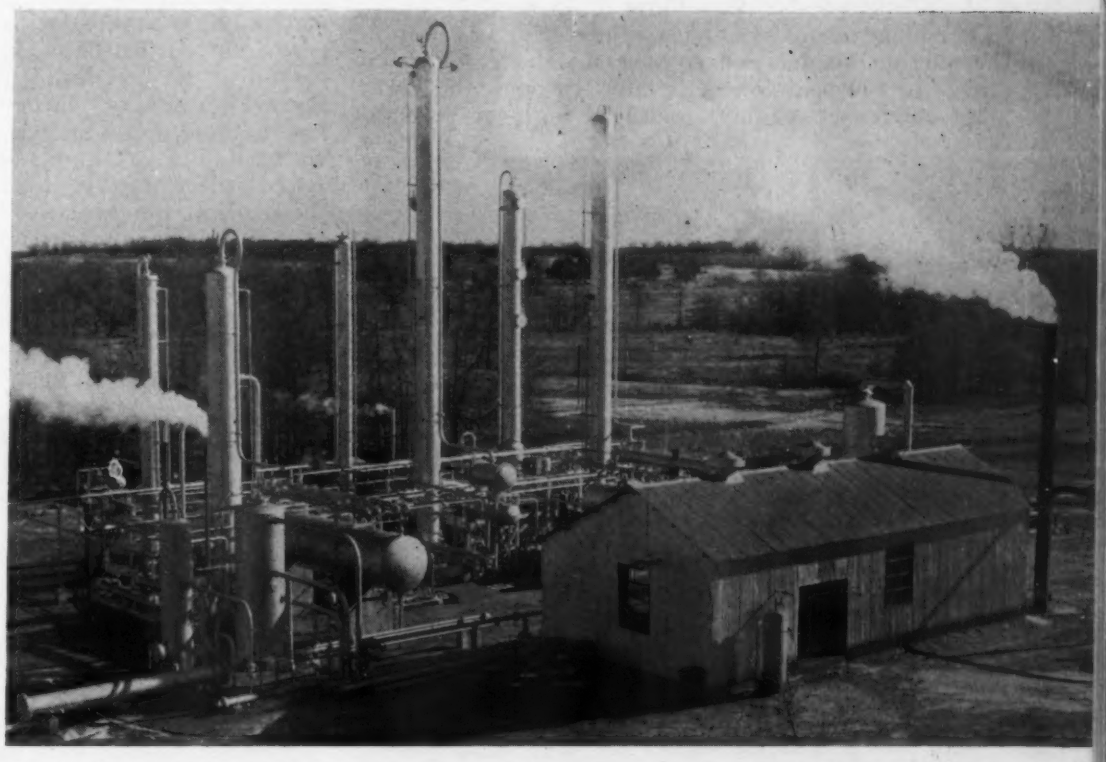
All of the materials used in the illustrations above are produced from hydrocarbon gases. Flow sheets could be prepared showing the reactions of the same chemicals with ketones, acetylene, hydrogen cyanide, ammonia and other reactive chemicals. A very wide variety of compounds could be prepared by using these additional materials.

The relatively short history of commercial organic synthesis has not induced prolific exploration into the multitude of product possibilities. Commercial encouragement is lacking in many instances, since the present basis for organic chemical development usually depends on the industrial demand. Demand hardly ever reaches real significance until the products enter the low-cost market.

Small Gasoline Plant Utilizes Waste Gas for Economic Operation

CHARLES A. SCHWARTZ, Consulting Engineer

IN RECENT years the trend in the natural-gasoline industry has been toward the building of larger plants, resulting in increased neglect of smaller fields. This trend started during a period of unstable and sometimes disastrous prices for natural gasoline, coupled with low demand and unstable prices for butane and propane. The war period with the material shortages, government controls, etc., accelerated the trend toward the larger plant. The same period saw development of larger cycling operations, and the revision of existing plants for higher extraction efficiencies and multiple fractionating processes. The labor and material situation of the postwar period has retarded development of practically all but the larger projects.



General view of Caska Corporation's gasoline plant at Quitman, Texas. Towers, left to right, are the utility still, main still, reabsorber, depropanizer, main absorber and stabilizer. Compressor building in foreground.

With the postwar conditions of increased demand for LPG products, increased demand for natural gas, increased recognition of the value of repressuring, and the steady pressure of gas-conservation groups, the need and possibility of building small plants that will show a reasonable economic return is growing.

Such a plant was built for Caska Corporation of Houston, in the center of the Quitman oil field, Wood County, Texas. The separator gas processed in the plant is from wells producing in the Paluxy zone. The wells are drilled on 40-acre spacing, in the field which is about 4 miles long and a mile wide. The crude oil has a gravity of from 41° to 45° API with a low (180) gas-oil ratio.

Design Considerations

The small plant described herein is of interest, not only for the economic return, but as one of the positive answers to gas conservation. At the time the plant was built there was no assurance of expansion. With a conservative estimated life of the field of 10 years, and with no increase in the production, it was estimated the plant would show a reasonable payout and return on the investment. Originally, the plant was designed for a maximum of 1,250,000 cubic feet of gas per day with a relative high absorption pressure, due to the small amount of gas to be handled and the higher extraction efficiencies obtained with a low oil rate. The higher absorption pressures permit greater flexibility on changes and operation on any future expansion program. All towers, line sizes, and most of the related equipment were made oversize, and are capable of handling approximately five times the expected nominal load.

Development of deeper production in the Quitman field and the gathering of additional gas from a number of small fields in a 10-mile radius presents opportunity for expansion of the present plant.

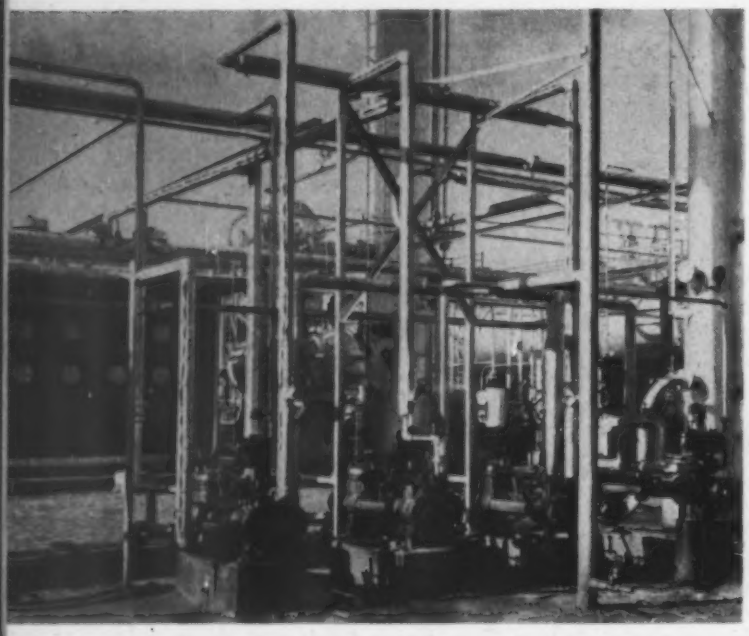
Fabrication of Vessels

All towers in the plant were either reworked, or fabricated completely on the job from used equipment. The depropanizer originally was 95 feet in height and was cut to 64 feet. The stabilizer was cut from 85 feet to 54 feet. The two sections from these towers and parts of three other towers were used in fabricating the high-pressure absorber. The still was made from two vessels of the same diameter and working pressure. The reabsorber, fat-oil vent tank, dehydrator, and several accumulators were made

from 23-inch diameter pipe. The 11.5 diameter by 42 feet butane-storage tank and 12 feet diameter by 38 feet gasoline-storage tank were fabricated on the job. Reworking and field fabrication of vessels not only lowered the original cost, but in addition permitted construction of larger capacity vessels at no extra cost. The critical material shortage and uncertain delivery schedules of shop fabricated vessels in certain instances dictated procedure. Flexibility in design permitted certain deviations from the original plan. These deviations for the most part were dictated by the availability of materials at the time of construction.

Gas Flow

The plant normally carries 5 inches Hg vacuum on the suction header and compresses the gas in two stages to 250 psig. The gas flows through the high-stage condenser accumulator to the high-pressure absorber. Dry gas from the absorber flows through a dry-gas scrubber to the plant fuel, instrument-gas dehydrator, and field residue systems.



Reflux and product pumps. Instrument panel in background.

Compressors

One of the interesting features of the plant is the use of steam-driven compressors. There are three horizontal twin gas compressors, handling an average of 1,100,000 cubic feet daily of 1.15 specific gravity gas. The compressor sizes are: 12 x 16 — 16 x 16 and 12 x 16 — 8 x 16; 10 x 14 — 14 x 14 and 10 x 14 — 7 x 14, and 8 x 12 — 10½ x 12 and 8 x 12 — 5½ x 12.

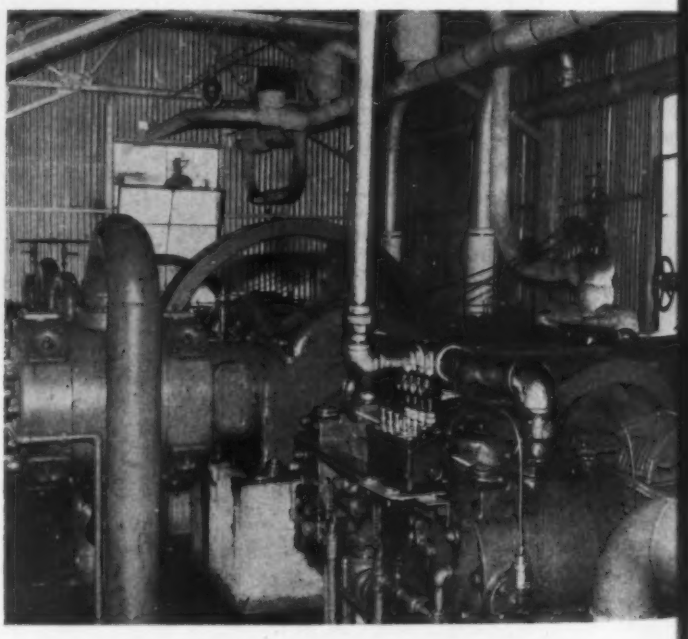
The advantages of steam compressors in this application are as follows: (1) Availability and low initial investment at the time of purchase. (These units were purchased as used equipment.) (2) Simplicity and flexibility of operation. As was expected in this small field, there are wide fluctuations in the gas load, suction pressures ranging from 10 inches Hg vacuum to 4 inches psig. The flexibility of the steam compressors permits them to carry this variable load without an appreciable change in speed. (3) Compactness of plant layout, due to elimination of the fire hazard inherent with internal-combustion compressors.

Obviously, there are certain disadvantages in the use of steam for the application. (1) Additional fuel is required per compressor horsepower, and (2) additional boiler capacity is required. A high shrinkage of volume in the gas treated due to products extracted, results in a fuel shortage on weekends and end of the month periods, due to present oil flowing schedules. Under normal operations, however, there is an excess of residue gas.

Residue Gas

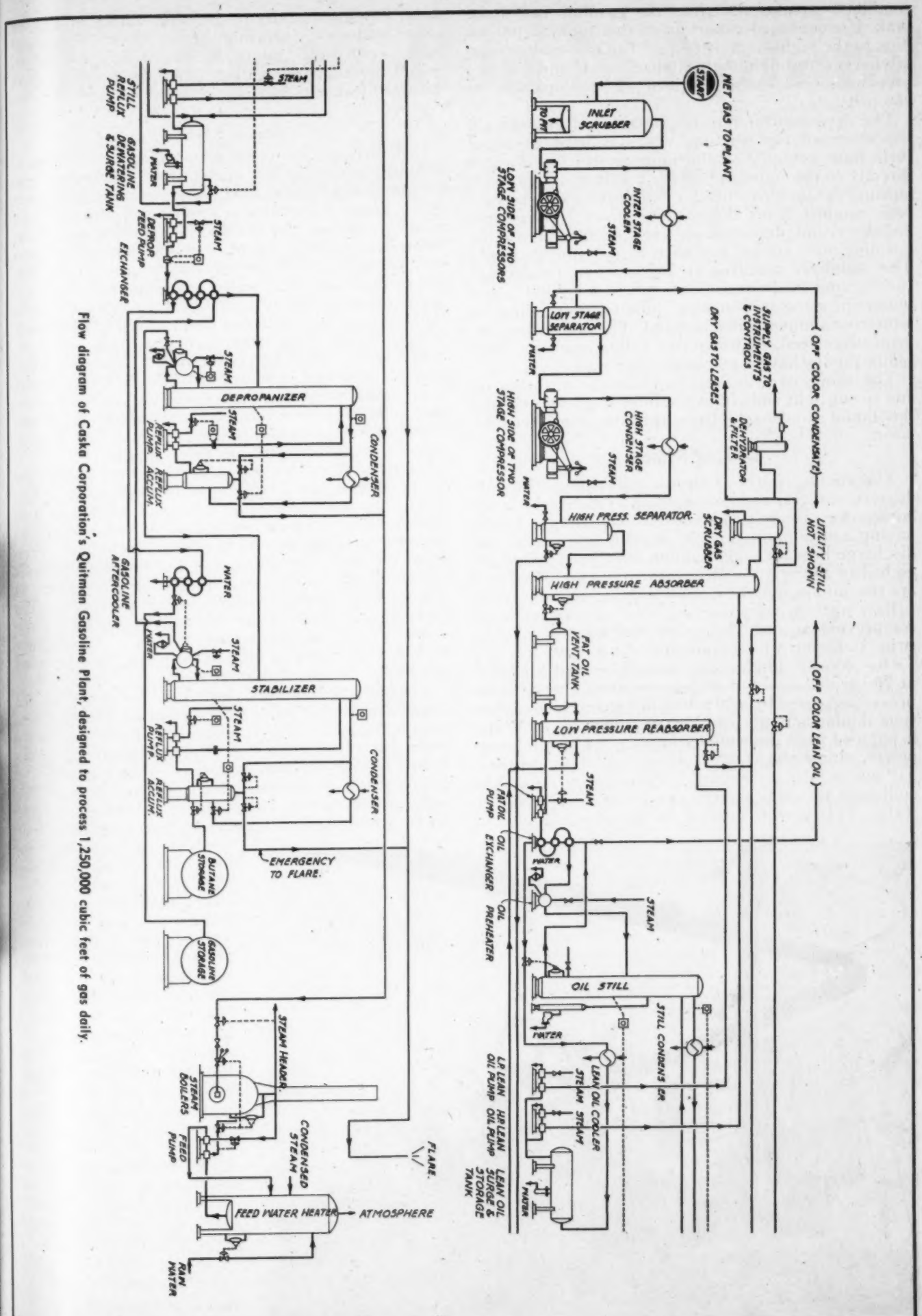
The fuel shortage and overage condition was eliminated recently by processing wet distillate gas. This gas was supplied as fuel for nearby drilling rigs. Distillate gas supply into the plant is controlled automatically, so that during the plant fuel-shortage period, more distillate gas is processed than is returned to the fuel line supplying the drilling operations. During periods when the plant has an excess of residue gas, it is supplied to the fuel line. Then, the wet distillate gas supply is reduced correspondingly. A control system regulates the incoming gas so that the plant no longer flares dry gas. All distillate gas is absorbed at line pressure. The volume of distillate gas processed averages about 700,000 cubic feet daily of approximately 0.8 gpm total liquid content.

The high-pressure absorber lean-oil pump discharges over the 250-pound absorber at the rate of 25 gpm. Fat oil from the absorber is flashed at 65 psig pressure. Flashed vapors, together with the uncondensed vapors from the gasoline rundown tank are reabsorbed at 65 psig in the reabsorber, which has a lean oil rate of 15 gpm. The flashed absorber fat oil with the reabsorber fat oil is pumped through the oil exchanger to the oil preheater, and then to the oil still, which operates at 100 psig. Stripped hot oil flows from the base of the oil still to the oil exchangers to the lean-oil coolers and to the lean-oil surge tank. The two lean-oil pumps and fat-oil pumps have a common spare.



Steam compressors provide flexibility necessary with varying flow of gas.

Condensed products from the high-stage accumulator at 250 psi is flashed to 100 psi, and the flashed stream commingled with the total overhead make of the oil still, downstream from the condensers. These



Flow diagram of Caska Corporation's Quitman Gasoline Plant, designed to process 1,250,000 cubic feet of gas daily.

combined streams feed into the gasoline raw-make tank. Uncondensed vapors from this tank at 100 psi flow to the reabsorber at 65 psi. The raw-make pump discharges through the stabilizer base and the depropanizer-feed exchanger into the depropanizer at 250 psig.

The depropanizer rejects propane and lighter as a vapor-discard top product, which is used as boiler fuel. Base product of the depropanizer is charged directly to the stabilizer, where a butane and propane mixture is taken overhead. Casinghead gasoline, as a base product from the reboiler flows through the stabilizer and depropanizer feed exchanger, to the gasoline after cooler, and then to the storage tanks. The stabilizer operates at 150 psi and produces a 16-26-pound-RVP base product and a 64-100-pound butane-propane mixture top product, depending on winter or summer sales demand. The still reflux, depropanizer feed, depropanizer reflux, and stabilizer reflux pumps have a common spare pump.

The utility still is used to clean the absorber oil and to cut light ends from the inlet scrubber drip and the liquid condensate from the low-stage accumulator.

Cooling System

The cooling water is circulated through the condensers, coolers, and compressors cylinders by a 40-horsepower steam-turbine-driven centrifugal pump, having a maximum capacity of 1400 gpm at 95 feet discharge head. It takes suction from the base of the eight-bay tower and discharges over the top. There are two pumps installed, one operating and one spare.

Raw water is supplied by a 550-foot well jetted by compressed air, which is delivered by a horizontal twin steam-driven compressor, 7 x 8 — 6½ x 8 and 7 x 8 — 6½ x 8 single-stage unit. The well kicks off at 70 psi and flows at 60-psi pressure. The air compressor is spared by a 12 x 12 x 11 vertical locomotive-type double-acting steam-driven compressor. Water is pumped from the well discharge pond by a 5-horsepower, single-phase, 220-volt motor-driven centrifugal pump into a 500-barrel water surge tank. The spill from this tank is used as cooling-tower-make-up water. This tank is located on top of a hill and the water flows by gravity to the plant, office, and to the boiler-feed-water aerating heater tank. The pond-water centrifugal pump is spared by a gasoline-engine-driven reciprocating pump, in case of power failure. Electric power is purchased.

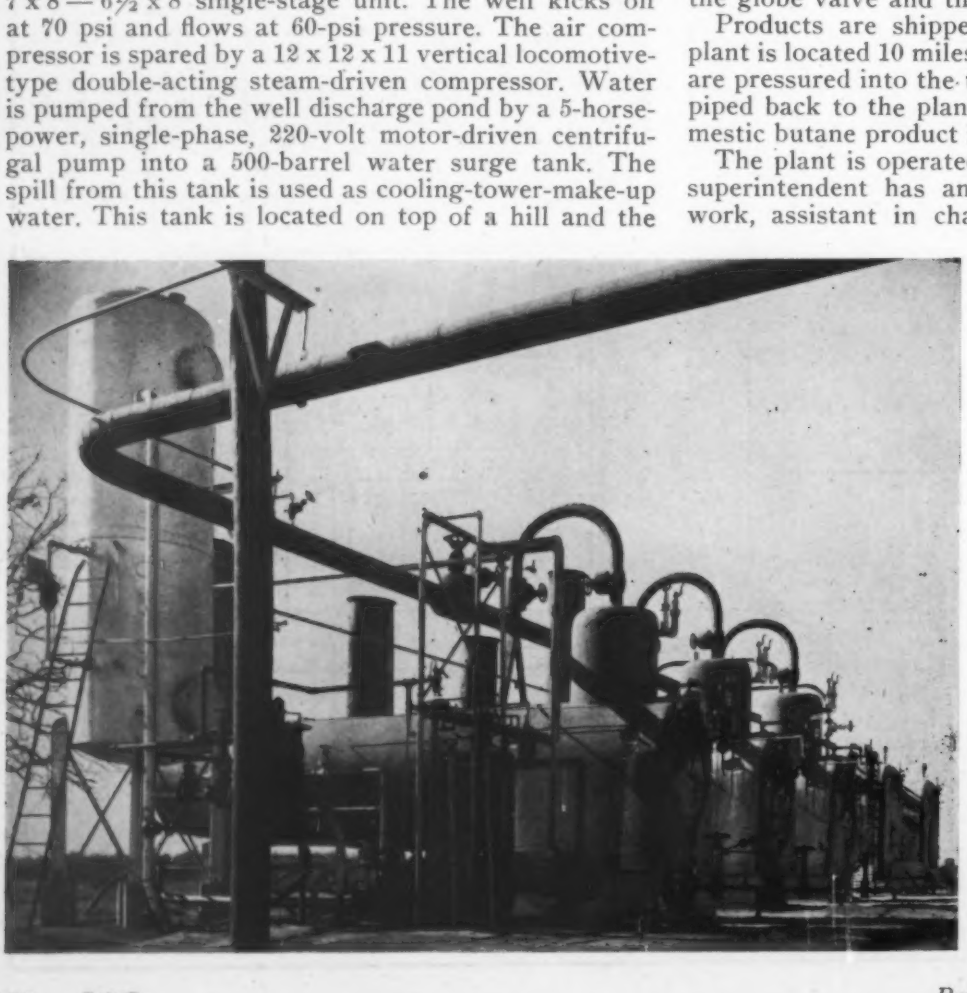
Steam System

Steam requirements are supplied by five 250-pound working pressure, 125-horsepower locomotive-type fire-tube boilers with four boilers operating and one spare. Steam is supplied at 200 psi and with full automatic control of the boilers. The feed-water heater receives condensed steam from the various reboilers and preheaters and the necessary amount of raw-water make up, for boiler requirements. The raw water is controlled by a liquid-level controller on the tank, and is deaerated and heated by the boiler-feed-pump exhaust. The boiler-feed pump and spare are controlled by a differential control valve, which maintains a 50-pound differential of feedwater pressure over boiler pressure. Each boiler has a liquid-level controller. The feed-water-control valves' diaphragm pressure is also transmitted to a snap-acting, manual re-set, emergency shut-off valve in the fuel line of each boiler. This shut-off valve is set to trip when the water level drops to the lower quarter of the gauge glass. The boilers also are equipped with high-level and low-level water alarms. Fuel-gas pressure is reduced in two stages with the boiler fuel-header pressure automatically controlled by the steam-header pressure, with a 10-pound limit control pressure, as a protection against over firing when starting up. The firing ratio of each boiler is manually set by a globe valve in the fuel line to each boiler. An emergency fuel-shut-off valve is also in each fuel line between the globe valve and the burners.

Products are shipped by truck inasmuch as the plant is located 10 miles from a railroad. The products are pressured into the trucks and kick-out vapors are piped back to the plant gas suction header. The domestic butane product is odorized when loaded.

The plant is operated with a total of 12 men. The superintendent has an assistant in charge of field work, assistant in charge of plant operation, four operators, four boiler firemen, and one man for day work.

The plant is on a 20-acre tract with a low hill in the center. The boilers, compressor building, processing area, cooling towers, storage tanks and truck-loading docks are located in a semicircle around the base and side of the hill. Each of the above operating areas are generously spaced to present the least danger from fire or explosion. The office and warehouse building and water surge tank are located at the top of the hill. All operating areas can be viewed from the office building.



Plant steam is generated in six oil-field-type boilers.

Fire Protection in Natural Gasoline Plants

C. L. HIGHTOWER, Safety Director, United Gas Pipe Line Company

THE modern natural gasoline plant represents a sizeable investment in process equipment, and because of the high pressures and temperatures at which process units are operated and the inflammable nature of the products handled, fire prevention and fire protection are highly important and should be given first consideration in the plant operating program.

Three elements are essential to fire. These are: fuel, oxygen and heat. When all three are present in the right proportions a fire and/or explosion will result. If any one is removed a fire or explosion cannot occur. All fire inspections should be made with the thought constantly in mind of keeping these elements separated, or removing one of the three when they are found in combination at the same point.

Hand fire appliances provide the greatest saving of property and life of all fire-protection equipment. Fifty to 90 percent of all the fires occurring in industry are extinguished with hand fire appliances without the necessity of calling the fire department.

The three major methods of extinguishing fire are cooling, smothering, and separation of flame from the fuel. By cooling, the generation of flammable vapors is stopped; by smothering, the oxygen supply which is necessary for combustion is cut off; and by separating the flame from the fuel, success is attained if no other source of reignition is present.

Fires are divided into three classes according to their fuel and the method necessary for extinguishing. All extinguishers approved by the Underwriters Laboratories are marked for the class or classes of fire they will extinguish.

Classes of Fires

Class "A" fires are fires that have materials such as wood, paper, textile, rubber, excelsior, rubbish, etc., as their fuel. These fires are extinguished by cooling with liquids or with extinguishers which use large percentages of water. Loaded-stream (instant) and foam-type extinguishers are recommended for Class "A."

Class "B" fires are fires in flammable liquids, greases, etc., such as gas, gasoline and oil. These fires are put out by blanketing or smothering to exclude the oxygen. Carbon-dioxide, foam and dry chemical-type extinguishers are recommended for Class "B."

Class "C" fires are fires in live electrical equipment. These fires require an extinguishing agent which does not conduct electricity in order to protect the operator against the danger of electrical shock. Carbon-dioxide type extinguishers are recommended for Class "C."

Proper Equipment

There is a place for most all types of extinguishers around the gasoline plant. Some units of the larger plants are sometimes equipped with enclosed automatic systems. Vapor-pump rooms, compressor buildings and storage and process vessels are examples of units that lend themselves to the enclosed automatic type of protection. The wheeled engine and the portable hand extinguisher are the most generally used, however. Even in larger plants where enclosed systems are practicable for use at given units, the portable extinguisher must still be relied upon for fighting certain types of fires and for emergency use at scattered and isolated units.

Some mention should be made of the use of water as a fire fighting agent. In some industries, notably the electrical, extensive use has been made of water-fog as an extinguishing agent. The specially designed water-fog nozzle sprays a fine mist of fog which serves to displace the oxygen in the atmosphere and the fire dies out because oxygen is not sufficient to support combustion. The water also has the effect of cooling hot metal or any wood embers that might be present.

The primary advantage of the water fire system in the gasoline plant is to provide water in sufficient quantity and pressure to cool off vessels and prevent boil-overs and the spread of the fire area. A solid stream of water directed on a burning gasoline spill or into a burning vessel of gasoline or other petroleum products will only tend to scatter the fire.

Maintenance of extinguishers cannot be stressed too greatly. There is a tendency to assume that because an extinguisher has nothing to do until a fire occurs, it does not require attention. Inasmuch as chemicals, pressure, mechanical parts and other features having a bearing on proper operation are all subject to the ravages of time, tampering or neglect, it is obvious that frequent maintenance is very important to proper performance.

Having an extinguisher that will perform as intended, another important item is its effective application. Regardless of its availability, condition, or potential effectiveness, all may be lost by improper application. Furthermore, additional hazards may be created, including danger to the operator.

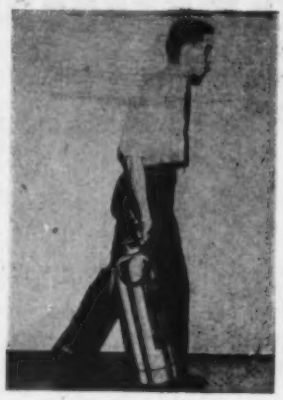
(Succeeding pages give some important pointers on use and maintenance of fire equipment for plant protection.)

Use and Maintenance of SODA ACID and FOAM Fire Extinguishers

Use Soda Acid Type on Class "A" Fires (wood, rubbish, cloth, paper, etc.) only. The soda-acid type employs wetting and cooling to extinguish fire. Foam Type is effective on Class "A" Fires and also on Class "B" Fires (gasoline, oils, grease, etc.). Foam covers the fire with a blanket effect and smothers by shutting off supply of oxygen.

1. Grasp hose close to nozzle between thumb and index finger of right hand.

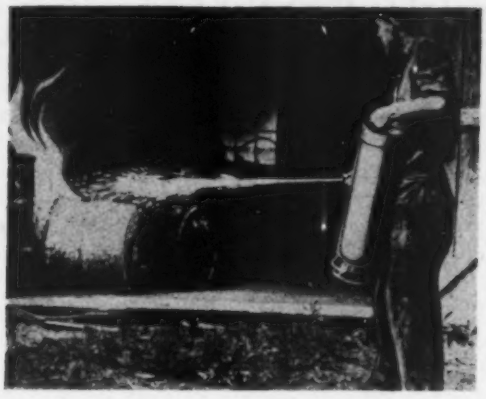
2. Holding hose, grasp ring cap while left hand grasps bottom. Lift extinguisher from bracket.



3. Carry extinguisher by ring cap in upright position to fire. Keep firm grip on nozzle.

4. Discharge by inverting extinguisher, holding by bottom handle. Hold nozzle firmly. Keep safe distance from fire. If outdoors, approach from windward.

5. Attack the base of the flame and work upward. Walk around fire, carrying extinguisher by bottom handle.



On Class "B" Fires (oil, gasoline, grease, etc.) direct foam against inside wall of container, just above burning surface. Avoid agitation by allowing foam to drop lightly on surface. Walk around fire. If fire is on floor or wall, stand back and allow discharge to fall lightly, preventing spread of flames. On Class "A" fires (wood, rubbish, cloth, etc.) discharge foam at base of flame and work upward.

6. Clean and recharge immediately after use. Discharge and recharge annually.

7. Inspect every six months. Check hose and nozzle for obstructions, check screen, acid bottle and stopple, cap gasket, and relief hole or holes.

8. Post information, date and sign tag on extinguisher. Replace tag when lost or damaged.

Soda-acid extinguishers are not suitable for Class "B" Fires (oil) and should never be used on Class "C" (electrical equipment) as the liquid is a conductor of electricity. Protect from freezing caps.

Foam extinguishers should not be used on Class "C" Fires, as they will conduct electricity. Protect from freezing.

Use and Maintenance of **CARBON DIOXIDE** (Liquid) Fire Extinguishers

Use on Class "B" Fires (gas, oil flammable liquids and other petroleum products) and Class "C" Fires (electrical equipment).

Recommended for use at the following locations: Trucks, boats, loading racks, compressor stations and pipe line repair equipment.

1. Remove from rack and carry by handle in upright position.

2. To operate, set cylinder on ground, remove horn and hold grip firmly with one hand. Pull pin and open valve with other hand. Small units may be held in one hand and operated with other.

3. For fires on flat surfaces, direct discharge at nearest edge of fire and, using sweeping side-to-side motion, extinguish nearest section first, then follow up fire. To conserve gas, close valve temporarily when moving around fire. Continue discharge after fire is out to avoid reflash.

4. For fires in small containers of flammable liquids, approach as closely as possible from windward side, and rest discharge horn on edge of container. Direct gas on burning surface at base of flame. Avoid blowing fluid from container. CAUTION: Maximum discharge range eight feet.

5. For electrical fires, aim discharge at base of burning insulation. CAUTION: Maximum discharge range eight feet.

Inspect and weigh extinguisher every three months. Recharge in weight loss is 10 percent or more below weight stamped on extinguisher. Check horn and hose for obstructions. Check wire seals and replace when broken or indication of tampering is apparent. Recharge after each use.

This extinguisher must be returned to the manufacturer for recharge. Keep nearest recharge station address posted on inspection tag. Record necessary information on tag and sign after each inspection. Replace tag when lost or damaged.

Annual recharging is not necessary as the contents do not deteriorate. CAUTION: This type extinguisher should not be used on Class "A" Fires (wood, trash, etc.) as they have glowing embers. Water or other approved liquids are best extinguishing agents for Class "A" fires.

Use and Maintenance of **DRY CHEMICAL** Type Fire Extinguishers

OPERATION

Keep extinguisher in upright position. Break wire seal holding lock pin and remove pin. Open valve to the left (left-hand threads). Point nozzle at base of flames, press nozzle release and sweep powder along base of flame. Pressure cartridge must be replaced after each use of extinguisher or accidental breakage of gas seal on cylinder. At least one extra pressure cartridge should be available at all times.

MAINTENANCE

Inspect every three months.
Remove and weigh pressure cartridge. If weight loss is more than one ounce, replace with new cartridge. Weight of pressure cylinder is stamped on hexagon nut.
Check hose for obstructions, and see that powder is dry and flows freely from hose.
Check powder level in extinguisher and add when necessary. Replace caked or lumpy powder.
Replace wire seal on locking pin when found broken and after each use of extinguisher. Record necessary information on tag after each inspection and sign. Replace tag when destroyed or damaged. Annual charging is not necessary as the contents do not deteriorate.

This type extinguisher should not be used on Class "A" Fires as they leave glowing embers. Water or other approved liquids are the best extinguishing agents for Class "A" Fires.

Application of Automatic Controls to Gasoline Plant and Refinery Processing

E. H. REYNOLDS, WILLIAM TROUTMAN and GORDON LAWN
Richfield Oil Corporation

IN ADDITION to theoretical considerations of automatic process control this paper presents actual accomplishments in operating units of both natural-gasoline plants and refinery-fuel-gas systems.

The paper was presented at the February, 1947, meeting of California Natural Gasoline Association, but was not ready for publication until April 1, 1947.

AUTOMATIC controls have been applied to gasoline plant and refinery processes with resultant improved products, and at lower cost. If a theoretical process could be set up in which all the factors affecting the process, such as feed composition, heating and cooling media, rate of heat transfer, etc., remained absolutely unvarying, the process could be established and would thereafter continue without change. Need for application of automatic controls, therefore, arises as a means of preventing variations of the factors affecting operation, or to compensate for their actual variation through control of suitable variables of the process. Thus, if variations in steam-line pressure supplying a fractionation-column kettle were upsetting operation, automatic control could be applied to control the steam pressure, or possibly to control the kettle temperature, and thus compensate for the variations in pressure.

Usually it is not possible to set up a control which can directly measure and control the product required, so the controls are set up to measure and control temperature, pressure, rate of flow, and liquid level, chosen in such a manner that their control will result in satisfactory operation with resultant production of the required product. For example, in controlling the operation of a deisobutanizer, it is not generally possible to analyze the products automatically with great speed and thus apply the automatic control based on this measurement. Instead, a suitable temperature in the column may be chosen which, at a given pressure, gives good indication of the product being produced, and the control applied to this temperature. Control can be applied to any suitable variable of a process which can be readily and rapidly measured, and which would aid in the overall control of the process; such as specific gravity, pH, color, etc., as well as the more generally used temperature, pressure, rate of flow, and liquid level.

Three Problems

Successful application of automatic controls involves the solution of problems. The first of these is rapid and accurate measurement of the variables of the process to be controlled. Successful control is impossible without dependable measurement. The second problem is rapid and precise application of corrective measures so as to maintain the controlled variable at the desired point with minimum deviation. The third problem is the selection of suitable variables of the process. Solution of the first two problems is contained largely in the control instrument.

There are two general systems of control in industrial service: electrical and pneumatic. Pneumatic control is more modern and presents several advantages. Its initial cost is low, it is flexible, servicing is not complicated, and it presents no fire hazard. For this latter reason, probably more than any other, it appeals particularly to the petroleum industry, and is in such common use in this field that the terms "control," "controller," etc., appearing in this paper will be considered to apply to the pneumatic system only.

In choosing an instrument for a control job, it is necessary to consider its characteristics first as a measuring device, and second as a controller. The subject of measuring devices is too broad to be covered adequately in this paper. No attempt will be made to do more than note the more common types along with some of their outstanding characteristics and limitations.

Measuring devices can be noted as follows:

Temperature

Pyrometers—include millivoltmeters and potentiometers. Both have as a primary element a thermocouple made of two dissimilar metals welded together at one end. The most common metal combinations in use as thermocouples in the refinery are iron-constantan with a normal range of O-1500° F. and chromel-alumel whose normal range is 0-1800° F.

A high degree of accuracy is obtainable with potentiometers. It is possible to have them check within ±0.1 percent of total range. They make very satisfactory controllers. This is especially true of the continuous balance type, since temperature changes are recorded without delay.

Thermometers—Bulb-capillary type thermometers are widely used as they are generally less expensive than pyrometers. They are commonly classified as follows:

Class I—Liquid filled, other than mercury. Work on principle of volumetric expansion. Evenly graduated chart. Temperature limit 400° F.

Class II—Vapor tension. A change in temperature causes a change in the vapor pressure of liquid filling medium. Unevenly graduated chart. Temperature limit 600° F.

Class III—Gas filled. Volumetric expansion of gas. Evenly divided chart. Temperature limit 800° F.

Class IV—Mercury filled. Volumetric expansion of mercury. Evenly divided chart. Temperature limit 1200° F.

Ambient temperature changes along the capillaries

of these systems cause errors, except in Class II, where they are minimized. Compensators are necessary to overcome this effect, especially where long capillaries are used. This type thermometer should not be used as a controller when capillaries are over 200 feet in length, since lag is too great.

Pressure

Pressure measuring devices are distinguished by the type of primary element employed—Bourdon tube, spiral, helix, and metallic bellows. Usually the Bourdon tube is used for high pressures, the spiral or helix for intermediate pressures, and the metallic bellows for low pressures. Location of pressure taps should be given some thought, remembering that the primary element will measure any static head present in addition to vessel pressure.

Flow

The differential type flowmeter with an orifice plate for a primary element is the standard means of flow measurement in the petroleum industry. There are two general classes in use, the mercury manometer, and the sylphonic bellows. The former is the older and by far the more common type.

The differential flowmeter is readily adaptable to control purposes.

Liquid Levels

Liquid levels fall into two general classes, float type and differential type. The first includes the recently developed displacement-type liquid level. The differential type is an adaptation of the familiar differential flowmeter.

It is common practice to let the level of the liquid swing between certain limits rather than holding it to a fixed point. This permits the addition or withdrawal of liquid from the vessel at a more gradually changing rate and may cause less upset in the process.

In the study of a control problem, consideration must be given to the type of control element best fitted for the particular job. There are available:

(1) The On-Off Controller in which the control valve is either wide-open or closed, depending on the position of the variable with respect to the control point. It has some application in batch processes.

(2) The Narrow Band Proportional Controller in which the control valve moves with the pen as it deviates from the control point, but at such a rate that a pen movement of from 1 to 10 percent of the chart range, depending on the throttling range setting, will move the valve from a closed to a wide-open position or vice versa. This control is useful in processes having very small time lag. It is designed to check the process deviation, but will not bring it back to the control point.

(3) The Wide Proportional Band Controller is the same as the Narrow Band Controller, except that it allows a choice of throttling ranges of from 1 to 150 percent.

(4) The Proportional Controller with Automatic Reset in which the addition of automatic reset to the Wide Band Proportional Controller makes it an element that will not come to balance until it has changed the air to the control valve sufficiently to bring the variable back to the control point. It is adjustable so as to set the rate at which the process variable will be returned to normal setting.

In many operations, control is improved by the inclusion of a valve positioner. This is true whenever conditions arise whereby the control valve does not readily respond to small changes in air pressure. They function by continuing to increase or decrease air pressure on the valve diaphragm until the valve assumes the position required by the primary control.

Lag

Consideration must be given to the ability of the measuring element of any controller to quickly detect a variation in the controlled medium and to actuate a mechanical device to correct this variation without undue loss of time; otherwise lag is introduced into the process. Lag is a term used to indicate the time interval between a process change and the repositioning of the final control element as a result. This can be broken down into (1) process lag, and (2) measurement-control lag. Lag is the principal bugbear of good control and its effect must be minimized whenever possible.

In order to fully appreciate the number of sources of lag in a control system, let us consider a bulb type temperature controller. A change of temperature outside the thermometer bulb is transmitted through the bulb wall and into the filling medium. This increases the pressure in the bulb, this pressure travels through the capillary to the spiral, moving the spiral. The spiral works through the control mechanism sending out a new air pressure through the connecting tubing to the control valve. The control valve changes the quantity of controlling medium. These steps follow in sequence, and although they overlap to some extent, there is, of necessity, an unavoidable time-lag. This inherent lag is not the same for all types of measuring devices, nor is it the same for various makes of pneumatic systems. Consideration should be given to this when planning the control of a critical process.

At any of the numerous steps between the variable change and the final control element, sufficient lag may develop to greatly impair the usefulness of a controller. Causes of lag in a temperature-controller might be:

The bulb or thermocouple is poorly located in the equipment.

The well is too heavy, or of poor thermal transmission qualities, or dirty.

There is an air space between the bulb and well.

The bulb is not fully immersed.

The capillary is too long.

There is loss of sensitivity in the measuring element.

There is lost motion or friction in controller elements.

The penumatic pilot has low capacity.

There are air leaks in pneumatic system.

Control valve action is sluggish.

Recently a study was made of the lag characteristics of copper tubing to determine the relative effect of tubing sizes and lengths. The test covered ¼-, 5/16- and ⅜-inch tubing in lengths of 50 to 300 feet by 50-foot increments. To simulate operating conditions, a control valve with pressure gauge was attached to one end of the tubing. The controllers were set to deliver 15½-pound air, and a cock was introduced between the controller and tubing. The time interval between opening the cock and the showing of 15 pounds at the control valve was clocked. Four standard controllers were used. One of the controllers developed less lag in the 5/16-inch tubing than in the ¼-inch, being 10 percent faster for 50 feet and 5 percent faster for 300 feet, it was slower when tested with ⅜-inch tubing. The other three controllers made their fastest delivery through ¼-inch tubing for all lengths tested. No two controllers delivered air at the

same rate, and they held their relative positions throughout the tests, the one being fastest for any length of any diameter tubing was fastest for all lengths and all diameters. The relative delivery rate of the fastest and slowest controllers was about 6:1.

Flow Control

With the control instruments that we have to work with in mind, let us now turn to the problem of selecting the variables to be controlled, and go to

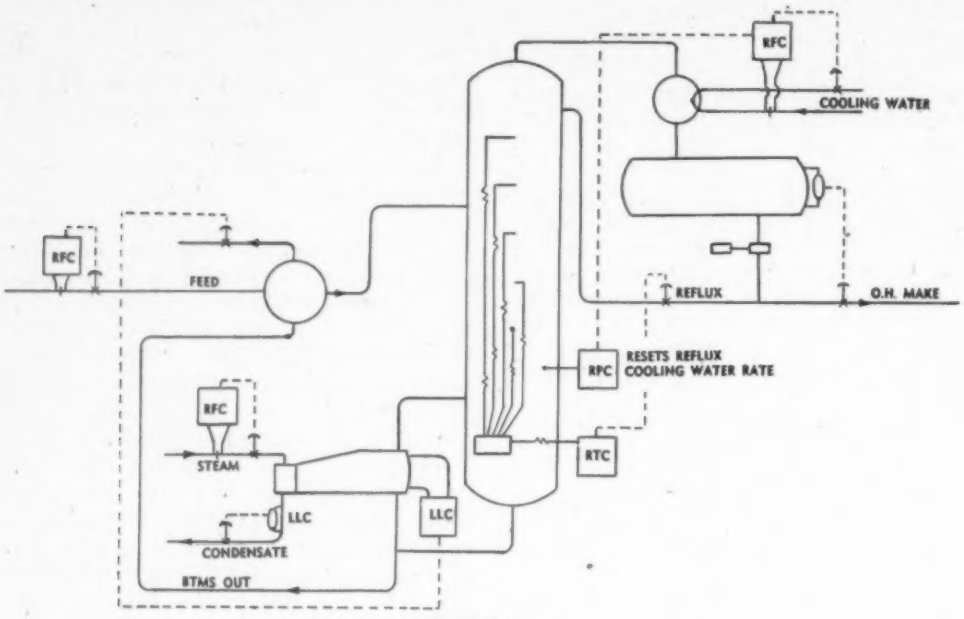


FIGURE 1
Deisobutanizer

Figure 1. This process is the operation of a deisobutanizer column. Feed to this column has approximately the following composition:

Propane—1 to 2 percent,
Isobutane—6 to 10 percent,
N-butane and heavier—88 to 93 percent.

The operation desired is production of an overhead containing a minimum of n-butane, while very low loss, say of not over 0.1 percent of isobutane is permitted in the bottoms.

The factors affecting the process that must be controlled or compensated for, are:

Feed composition and rate.
Cooling-water temperature and pressure.
Steam temperature and pressure.
Tower heat loss.

The flow controller on the feed controls the feed rate directly, since variation would throw an additional load on all ensuing controls.

The flow controller on the steam to the reboiler is used to minimize fluctuations of heat input due to variations of steam pressure. A pressure controller on the reboiler steam pressure might be preferable to the flow controller used, but the flow controller was on hand and was used since variations in heat input are compensated for by other controls.

The liquid level control on the reboiler condensate is used to release condensate from the reboiler at whatever rate formed.

Temperature control is used to control the temperature on one of several points in the tower, since at constant pressure this will maintain the products close to that desired, despite normal variations in feed composition, heat input, and tower-heat loss. Location of the point at which the temperature in the tower is controlled must take into consideration the particular type of operation desired. If the temperature at the bottom of the tower were controlled, variations in the heavy material in the feed stock would affect this temperature without necessarily reflecting a change in the overhead product. If the top temperature of the tower were controlled, the varying propane content would have an appreciable effect on this temperature, and so the n-butane content would vary inversely. By selecting an intermediate point, it is possible to minimize the effect of variations of propane in the overhead and still have a point which will vary with changing n-butane content of the overhead. The actual point used is the 53rd tray from the bottom in a 70-tray tower. This temperature is controlled through varying the reflux to the tower, since reflux enters at a point near enough to that of temperature measurement that a reasonable time-lag will result.

The pressure-control tap should be as close as possible to the point at which the temperature is controlled, since a given temperature controls the product produced only at a given pressure. The pressure control operates by controlling the cooling water through the overhead condenser, thus varying the condensing rate on the overhead. In order to compensate for variations in cooling-water pressure without first requiring a change of pressure in the tower, a flow controller is used in the overhead-condenser-water system. The tower pressure controller resets the control index of this flow controller. This is necessary in the particular application because of other equipment taking off of the same cooling-water system, and in cutting between isobutane and n-butane with a difference in boiling point of 19°, very close temperature and pressure control are required to maintain product composition.

Liquid Level

Liquid-level control on the overhead controls the overhead make and thus adjusts for the varying ratio between overhead and bottoms due to feed-stock-composition change. The liquid-level control on the reboiler serves the same purpose, but it might be noted that this is a differential type controller having proportional control with automatic reset, and is used

because it is desired to maintain the bottoms flow at as constant a rate as possible and still keep the level within limits. This is necessary on account of the bottoms heat exchange with the feed stock so that the temperature of the feed to the tower will remain as constant as possible.

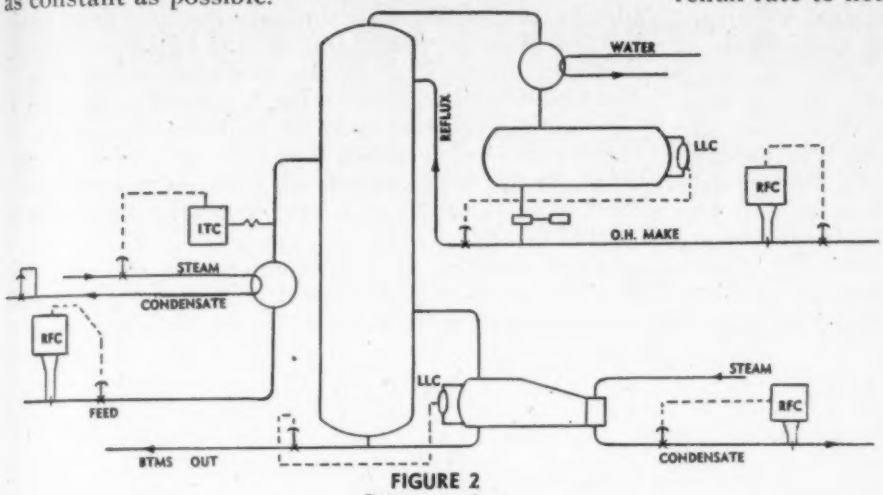


FIGURE 2
Deisopentanizer

Figure 2 represents application of automatic control to a deisopentanizer tower. Feed to this column is of approximately the following composition:

N-butane—0 to 1 percent.
Isopentane—45 to 49 percent.
N-pentane—50 to 54 percent.

It is desired to produce an overhead containing as much of the isopentane as possible with not over 7 percent n-pentane.

It should be noted that both the overhead and bottoms products desired are nearly pure products, hence have little change of boiling point with the amount boiled off. It is not practical to control this operation by the use of a sufficiently sensitive temperature and pressure control at a mid-point within the column as was illustrated in Figure 1, due to the very small difference in boiling points (approximately 9°) between the isopentane and n-pentane. The temperature at the point of measurement would have to be held to within a fraction of 1°. Instead, advantage is taken of the fact that the feed composition changes slowly and the tower is set up to make a given split between overhead and bottoms, this split being varied in accordance with tests on the overhead and bottoms products.

The factors affecting the process that must be controlled, or compensated for, are:

Feed rate.
Cooling-water temperature and pressure.
Steam pressure and temperature.
Tower-heat loss.

The flow controller on the feed controls the feed rate directly. The temperature controller after the feed pre-heater controls the heat input with the feed, and compensates for variations in steam pressure to the preheater. The flow controller on the condensate from the reboiler controls the heat input through the reboiler, and compensates for changes in steam pressure to the reboiler. The flow controller on the overhead product sets the split between overhead and bottoms. The liquid-level control on the overhead accumulator compensates for any changes in heat input or cooling by varying the reflux rate to hold this level. The liquid-level control on the bottoms controls the bottoms make as available, while maintaining a liquid level in the reboiler.

Refinery Fuel Gases

Figure 3 represents application of automatic controls to the problem of maintaining the pressure in the refinery-fuel-gas system by burning the excess gas under the boilers. In the refinery there are many processes consuming fuel gas and many processes releasing gas into the fuel system. Due to variations in operation, the balance on fuel gas in the refinery is always changing. Before the present controls were set up, commercial gas was available to take this swing. When this was no longer available, it became necessary to either waste large quantities of gas during periods of excess, or to arrange to satisfactorily use this gas in varying quantities as available. Many fluctuations in this gas supply are too fast to permit manual adjustments of fuel gas consumption in the refinery.

Referring to Figure 3, a pressure controller on the fuel-gas line controls the pressure on this line by releasing gas to the burners under the boiler. The flow controller on the steam from the boiler controls the fuel oil to the burners in order to compensate for variations in the amount of gas burned, as they tend to cause a change in the steam flow. In order to compensate for variations in the fuel-oil-supply pressure without first causing a change in the steam flow, a pressure controller is used on the fuel oil to the burners. The flow controller on the steam resets the control index of the pressure controller on the fuel oil. The flow controller on the fuel gas to the boilers serves to limit the amount of gas which can go to the boilers, since it will not permit passage of more gas than it is set for at the maximum orifice-plate pressure, which would be the fuel-gas-system pressure.

The above control system will accommodate

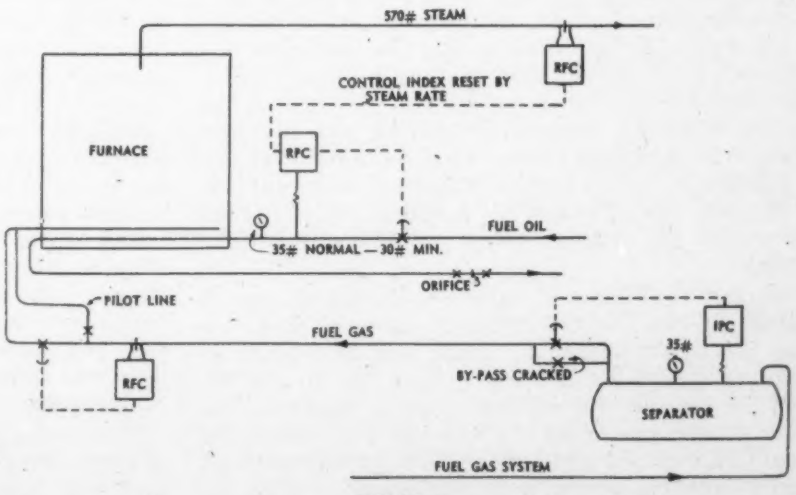


FIGURE 3
Refinery Fuel System Pressure Control

changes of rate in the available fuel gas of approximately 150,000 cubic feet per hour, making this change if necessary in 2 to 3 minutes without noticeably affecting the output of the boilers.

The above fuel-system-pressure-control arrangement is sufficient to accommodate most normal changes but is not sufficient to take care of surges sometimes caused by upset operation, particularly of a cracking plant. These plants can very suddenly change from a net producer of fuel gas to a very large consumer of gas when operation is badly upset, say by a power failure. At a time such as this, if the large quantity of fuel gas required were not immediately available, temperatures would continue to fall and operation would be lost. There are also times due say to a sudden increase in fuel gas from the vapor-recovery system, when the excess fuel gas is temporarily more than can be burned.

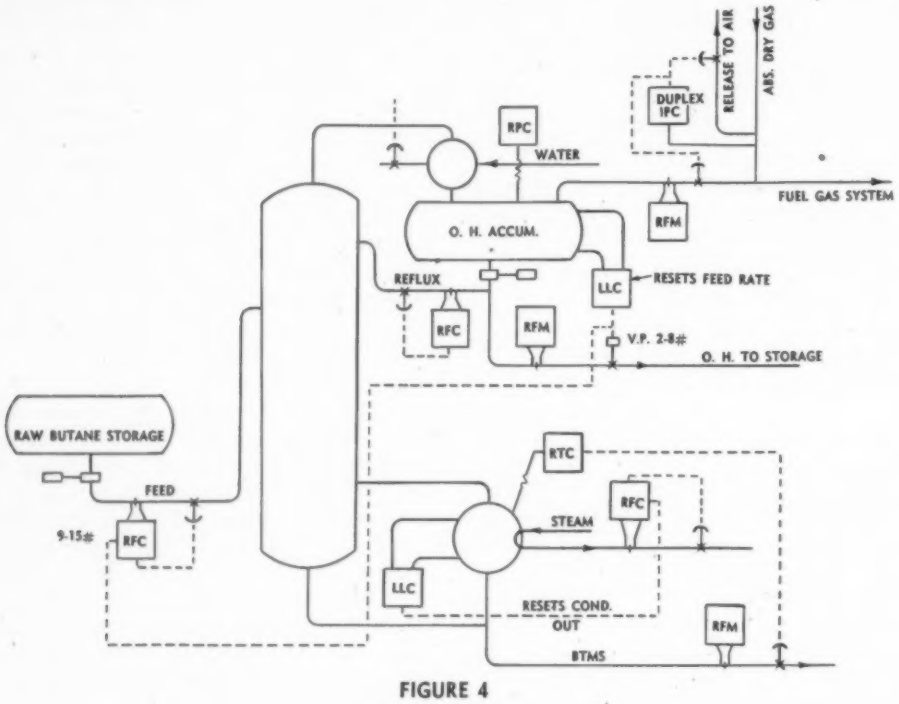


FIGURE 4

Fuel System Makeup

Figure 4 shows an automatic means of limiting variations of fuel-gas pressure by use of a duplex pressure controller which will allow make-up gas to go to the system when the pressure drops to a set point, or will vent gas to the air if the pressure rises to a predetermined point. In order to automatically have gas available for make-up into the fuel system, a stabilizer is kept in operation, feeding n-butane stock, the overhead from which returns to the feed tanks while the bottoms product containing any material heavier than n-butane is discarded to other tankage. The operation desired on this stabilizer is the supplying of fuel gas containing nothing heavier than n-butane, at any time, and in whatever quantities required.

Referring to Figure 4, when no make-up gas is required, the feed to the stabilizer is controlled at a pre-set minimum which will allow the feed pump to operate properly. As a level is built up in the reboiler, the reboiler liquid-level control increases the flow of condensate from the reboiler by setting the control index on the condensate-flow control until all material in excess of that required to maintain the reboiler level is boiled off back into the tower. The temperature control operates from the reboiler temperature and adjusts the bottoms rate to permit removal of all material heavier than n-butane. In order that the temperature of the material in the reboiler will indicate its composition, a constant pressure is maintained. The pressure controller which does this adjusts the water rate through the overhead condenser. Material not going out as bottoms will go overhead and appear in the overhead accumulator. The liquid-level control on the overhead accumulator adjusts the overhead make to maintain a level in the upper part of this vessel. The flow controller on the reflux to the tower maintains a set reflux rate sufficient to insure removal of the heavy material in the bottom product.

When make-up gas is withdrawn for the fuel system, the pressure controller decreases the amount of overhead condensation to maintain the set pressure. This results in the liquid level in the overhead accumulator starting to drop, causing the level control to decrease the amount of overhead returned to storage. The control valve on the overhead product is coupled with a valve positioner set to give full valve travel for a 2-8-pound range of the controlling air pressure. When the liquid level on the overhead accumulator has reached mid-point, the overhead valve is closed. If the gas release is greater than the minimum set feed will provide, the liquid level in the overhead accumulator will continue to fall and thereby increase the feed to the tower by raising the control index on the feed-flow controller. The control index of the feed controller was adjusted in our own shop so that it travels full scale for a 9-15-pound range of the controlling air pressure. However, index setting mechanisms are available which are adjustable both as to the air pressure at which motion starts and also as to the rate of movement with change of air pressure.

When the gas requirement for make-up to the fuel system decreases, the above process reverses itself and will return to the original operation or will stabilize itself at any intermediate point.

Basic Petroleum Chemistry For Refinery Employes

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Chapter X

THE REFINERY OF THE FUTURE

WHEN Harold Ickes was Petroleum Administrator For War, he paid this well earned tribute to the accomplishments of the petroleum industry. He said: "Since the Nazis and the Japanazis started on the rampage, the world of science and invention has accomplished what normally it would have taken 50 years to do. This is especially true of the American Petroleum Industry—. With the biggest order of its life to fill, they (American oil men) went to work with grim determination, and became, as much as any other factor, responsible for the means by which it (the war) will be won by the United Nations."

Certainly it would have taken many years of normal peacetime development to attain the degree of perfection found in some of the newer refinery processes, the more important of which have been briefly discussed in this series of articles.

Just what is ahead in refining is contingent on many unpredictable developments. Expanding research programs and the application of wartime developments to the future refining picture makes it difficult to evaluate present trends. It would be presumptuous for one to attempt even a mere outline of possible future developments in petroleum refining without the help of experts in various fields related to the industry. This chapter is therefore a composite of the views held by experts in the fields of economics, science and engineering.

Without doubt, the future will see gradual improvements in basic refinery processes as well as in the more complex chemical refining processes hurriedly pressed into use under the stress of war. Fifty years of petroleum technical progress compressed into the short span of about five years, will not result in technical complacency. As a matter of fact, throughout the industry there is evidence of unprecedented appropriations for new laboratories, pilot plants and technical personnel.[1,2,3,4] There is a much healthier respect for the value of research on the part of those who control the purse strings. Several petroleum organizations have announced their intention to at least double the scope of their present research activities. Despite this anticipated expansion, a relatively small percentage of total investment is being devoted to research and development. It is quite certain the future of petroleum refining will witness a considerably larger percentage of its total investment devoted to research facilities.

During the last year the volume of products from the petroleum industry increased to an all-time high, greater than during any war year, and more than 24 percent above the highest prewar peak.

Contrary to early predictions, which anticipated a somewhat gradual return to prewar demand, all refined petroleum products except aviation fuels have rapidly recovered postwar losses, and in some instances have considerably exceeded the prewar and also the wartime peaks. Crude-oil production in 1941 was 4,072,000 barrels daily. The peak war-time production in 1944 was 4,864,300 barrels per day. Last year the approximate daily production was about 5,075,000 barrels. The 1947 demand for petroleum seems certain to exceed many of the estimates made during and since the war.[5,6] World demand for crude oil, excluding Russia, is expected to increase to about 8,350,000 barrels per day by 1950[7] and at least 65 percent of that quantity will be consumed in this country.

There will be plenty of crude oil available, with larger volumes likely to be imported from abroad. There has been a decline in the production in some of the Mid-Continent areas which will result in the necessity of some refiners processing more marginal crudes of lower volatility and high sulfur content. The high-sulfur crudes are becoming less of a problem to handle because of progress in the practical application of corrosion-resisting alloys to refinery equipment, improvements in the efficiency of crude-conditioning methods, and improvements in distillate-treating processes.

The demand for aviation gasoline reached a peak during the war in excess of 500,000 barrels per day. This high demand is not likely to be reached again soon—certainly not before 1950, unless we again become involved in another war. However, with the increase in commercial air travel, air cargo, military aviation and private flying, the 30,000 barrels per day demand of the prewar period is expected to be expanded to about 115,000 barrels per day by 1950.

The development of jet-propelled planes is now placing upon the refiner the obligation to perfect suitable fuels much different in characteristics from those formerly used in internal combustion engines. The entire problem of aviation fuels is very much in a state of uncertainty, depending upon the outcome of the vigorous aeronautical research instigated by lessons learned during the war through experience,

and since the war through the confiscation of enemy scientific records and patents.

A new system of rating the anti-detonating properties of motor fuels is needed and will result from the present confusion over the several different octane ratings in use. The inadequacy of the 20-year-old octane scale for evaluating the modern super fuels was felt keenly by technical groups during the war. A "triptane number" has been suggested at the United States Bureau of Standards. The proposed rating would be in terms of blends of triptane and normal heptane. It will be recalled (see Chapter VI) that the hydrocarbon compound triptane, or 2, 2, 3 trimethyl butane, has an exceptionally high anti-knock rating, and the new scale would cover a range from below to above the normal ratings for present day fuels.

Motor Gasoline

If all of the excess postwar aviation gasoline were dumped into motor gasoline, it would result in an increase of only a few octanes, and the quality gain would be far too expensive for the highly competitive market. Some of the aviation units are much too costly to operate in a peace-time economy and will remain shut down indefinitely. Others are being converted into equipment which can be economically operated for the production of motor gasoline components.

Table 12 represents an estimate as to how the production from aviation units might be used to improve quality and augment postwar gasoline over prewar averages.[8]

TABLE 12

Estimated Proportionate Production of Postwar Gasoline Compared with Prewar Production

GRADE	NATIONAL POSTWAR AVERAGE		
	Percent Total Production	Octane Number	
		Motor	Research
Aviation	6— 8	100	.....
Premium	10—20	85—88	93—98
Regular	60—75	78—80	83—88
Third Grade	5—15	68—70	70—74

GRADE	NATIONAL PREWAR AVERAGE		
	Percent Total Production	Motor	Research
Aviation	2	91—100	..
Premium	10	80	86
Regular	78	75	80
Third Grade	10	64	65

Research octanes are expected to increase to a greater extent than Motor-Method values. This is expected because of increasing use of catalytic-cracking operation which result in the formation of higher concentrations of aromatic hydrocarbons than is possible with most thermal-cracking equipment. A wider spread between Motor Method and Research octane values for a given fuel results in smoother performance.

This is only a part of the picture as far as quality improvement is concerned. New equipment and new processes will enable the refiner to control desirable qualities better than was possible before the war. When the new light-weight automobiles, predicted for the future, begin to make their appearance, the refiner will be ready with the proper fuel. Future gasolines will be composed of mixtures of relatively few hydrocarbon compounds as compared to the gasoline we know today. The mixtures will be free of odoriferous compounds. There will be more miles per gallon available in the motor fuel of the future.

The demand for motor fuels has continued to increase in volume since V-J day and only a serious depression or the continuation of serious controversies between management and labor in all industries, particularly the automotive and petroleum industries, would cause a downward trend in the demand. C. L. Burrill, economist for Standard Oil Company of New Jersey, in a paper presented before the Petroleum Division of the American Institute of mining and Metallurgical Engineers, predicted a total demand of 862,500,000 barrels by 1950. This figure represents a 24-percent increase over the 1941 consumption.

Mid-Distillate and Residual Fuels

The mid-distillate fuels, including kerosine, domestic heating oils and diesel fuels, are prophesied to have a greater percentage increase than gasoline. Home-heating-oil demand is expected to increase 37 percent, and kerosine 7 percent within the next five years. The most phenomenal increase is expected in the field of diesel fuels. The demand for these fuels has been prophesied to increase 100 percent within the next five years, largely as a result of anticipated increase in the use of diesel trucks and diesel locomotives. Diesel horsepower in the United States increased from 5,500,000 in 1935 to 45,000,000 in 1946. In ten years it is expected that the railroads will be 40 percent "dieselized," and fuel requirements for this purpose alone would be on the order of 120,000 barrels per day.[9] At least some of the diesel fuels may come from natural gas through commercial application of the Fischer-Tropsch or Synthine process. Diesel fuels made by this process were employed by the Navy in its demonstrations at Annapolis last year and were found to be definitely acceptable or superior by almost every present standard. German diesel fuels made by the Fischer-Tropsch process are often in the range of 90 to 100 cetane number. It is logical to suppose that the increasing demand for mid-distillate fuels will cause the refiner to look to this process as a possible source of high-quality diesel fuels.

Income of the American farmer is at an all-time high, resulting in an insatiable demand by the farmers for mechanical equipment of all kinds, particularly tractors, which are heavy consumers of mid-distillates. A movement now under way proposes to increase the sale of both distillate fuels and residual fuels, in the city and on the farm, by concentrating on an advertising campaign to sell such fuels to the consumer on an annual contract basis, including burner service, at prices low enough to compete with coal and gas.[10] Between 1935 and 1945 home-heating units doubled from 1,000,000 to 2,000,000 units. The present home-building boom is increasing the number of oil-burning units at a rapid pace.

This great demand for mid-distillate oil will make it advisable for refiners to make use of larger volumes of recycle distillates from cracking operations in blends of diesel and domestic heating oils. Such blends will call for the use of additives which have been developed to prevent excessive gum formations. Other additives will be required to improve ignition qualities of diesel-fuel blends. The virgin distillates usually have good ignition qualities (cetane number) and ordinarily do not require such additives. These virgin stocks, however, will be needed in larger vol-

umes to meet the demand for thermal- and catalytic-cracking charging stock.

In 30 years the world's merchant fleets have changed from 4 percent oil-burning to 75 percent oil-burning, and the few remaining coal-burning vessels are taking on oil-burning units at a rapid rate.

Miscellaneous Petroleum Products

The preparation of many petroleum specialties, including special solvents, waxes and chemicals, is becoming a very important branch of refining. In Chapter VIII a number of petro-chemical processes were discussed. The manufacture of organic chemicals and chemical intermediates, numbering into the hundreds, is becoming more and more a part of the important function of tomorrow's refinery.

The manufacture of synthetic rubber faces an uncertain future. However the most economic manufacture has been found to be from butadiene made from petroleum. Even if the maximum quantity of natural rubber was made available in this country by the relaxation of government control, at least 200,000 long tons per year of synthetic rubber would be required. Two good arguments favor the continuation of government control. First, the government recognizes the need of keeping the young synthetic rubber industry active for military reasons. Second, the manufacturers of rubber goods favor the continuation of government control as a protection against the wild fluctuation in the price of natural rubber. Therefore, much more than 200,000 long tons of synthetic rubber per year will be required for two or more years, and refiners will be called upon to furnish the necessary butadiene and special solvents.

The national dollar-volume consumption of all types of plastics has grown rapidly in recent years. Planned facilities will bring the capacity to 3½ times the 1945 volume and 37½ times the volume of ten years ago. Refiners will share in this lucrative field.

While there is expected to be an increase in the demand for all kinds of lubricating oils, the chief concern of the refiner will be to improve on the quality of many existing lubricants. Emphasis will be given to the development of better lubricants for automobiles, aircraft and special intricate machinery. Better lubricants for jet propulsion, gas turbines and close-clearance engines of all kinds will enter more into the refinery picture. Solvent refining methods will be improved and expanded. Synthetic lubricants will be demanded for very special uses. Lubricants making use of such compounds as the hydrosilicons will become a manufacturing activity of the refinery of the future.

Atomic Energy

Several major oil companies are investigating the possible industrial application of atomic energy, but no one seems to be perturbed over the possibility of early competition of atomic energy with petroleum—at least not yet.

Such fantastic statements as "The Queen Mary can be driven to Europe and back with a piece of uranium the size of an egg" or "A piece of uranium the size of a pea buried under the doorstep will heat a house throughout its life time" have not caused petroleum technologists to lose sleep over the possibility of loss of maritime or domestic fuel sales. In fact, the natural-gas industry has heralded this possible source of energy as a safeguard against the ill effects of eventual depletion of its valuable products, and has used it as an argument against those who insist that the supply of natural gas is about gone and must be husbanded.

While it is estimated that atomic energy will be available for industrial application within the next five years, its application most likely will be in remote mining operations and irrigation developments far removed from serious competition with petroleum.[11]

One pound of unranium 235 will give, on fission, as much heat as 1,500 tons of coal worth between $7,000 and $20,000. From fragmentary data it is estimated that uranium costs about $22 per pound. However, the cost of equipment for processing it into useable energy is very high and only a very small fraction of the uranium can be fully utilized. Nevertheless, it has been estimated that with the developments now planned, in ten years atomic fuel might compete with coal selling at $10 per ton.

Unless someone can devise a cheap atomic-powered automobile or airplane to compete with gasoline, serious competition with petroleum is not expected for 25 years, and perhaps never. On the basis of these estimates, we must conclude that there is no imminent danger of atomic energy putting petroleum refiners out of business.

The status of atomic-energy utilization is summarized in the following quotation from a report of a large group of scientists advising the Secretary of War's Interim Committee on Atomic Energy:

"We have examined in some detail the technical problems of making available heat and power on the scale of present world consumption from controlled nuclear reactors. We see no significant limitations on this development, either in availability or in the cost of the fundamental active materials. We see characteristic limitations and characteristic advantages in atomic power which make us regard it in great measure as a supplement to existing sources, and an incentive to new developments, rather than as a competitor, let us say, to coal or to petroleum products We see no foundation in current science for the hope that atomic power can be effectively used for light, small portable units such as are required for aircraft and automotive transportation; but we believe that the development of rather large power units for heat and conversion to electrical energy is a program for the near future; that operating units which serve to demonstrate the usefulness and limitations of atomic power can be in existence within a few years, and that only the gradual incorporation and adaption of such units to the specific demands of contemporary economy will involve a protracted development."[12]

New Processing Equipment

New equipment added to refinery facilities during the war seems to have accelerated the demand for more of the same. The refinery of the future will become a combination oil refinery and chemical-manufacturing plant. Catalytic and thermal processes will be built for maximum conversion of crude oil into high-octane fuels or special organic chemicals. More refinery gases will be polymerized to higher-value liquids. Super-fractionating equipment which may employ 150 or more plates will be used for the separation of relatively pure hydrocarbons from petroleum mixtures. Improved extraction and solvent refining equipment will be used to improve upon the separation made possible by precise fractionation.

More and better instruments, making use of recently developed application of electronics to processing controls will be required for the flexibility which

will be more necessary in tomorrow's refinery. Demands for a larger variety of petroleum products are likely to fluctuate as a result of expanded research and development in the field of fuels, agricultural uses,[4] chemicals, lubricants, solvents, plastics, etc. The refinery must, of necessity, be extremely flexible. It must be capable of making a large variety of products demanded by an ever changing market. Refineries may become less competitive, with each refiner becoming a specialist in some branch of manufacture. Motor gasoline as it is known today will become a secondary source of revenue.[13] Profits will be found more and more in the sale of special organic chemicals, special solvents, lubricants and intermediates for a greatly expanded organic chemical industry.

Refinery equipment has always had a rapid rate of obsolescence, and as a result of expanded research facilities, processing equipment is certain to become obsolete even more rapidly in the refinery of the future. Different equipment will be required if and when it becomes necessary to supplement the petroleum supply with products made from coal, oil shale and agricultural wastes.

Refinery Personnel

One of the problems uppermost in the minds of refinery managements is the question of adequately trained personnel. The more complex refinery operations become, the more important it becomes for workers to be trained in the complexity of their respective duties. Training has to be extended into all branches of the refinery, and it should reach down into the laboring groups.

Progressive refineries saw this need emphasized during the war years and those which had not already established training facilities within the organization were quick to set up training departments aided by the educational institutions, government agencies and especially by industrial publications.[14,15] It is said that the technology of refining has become at least five times more technical than it was some 20 years ago. The process developments of the engineers and chemists are demanding specialized knowledge on the part of unit operators and their assistants. New metals, alloys and plastics are coming into refinery use more each day. Machinists must know how to handle them. New intricate machines require greater skill on the part of personnel engaged in the crafts and in general refinery-maintenance work. Skilled operators and craftsmen can not be acquired in the labor market because of limited avalability and because of agreements with labor groups to promote from within the existing organization when possible. Refinery organizations will have to see to it that men get the necessary training on the job.

The trends in refinery training are aimed at reaching every group from the laborer to the department heads. For those employes who are less disposed to the study of available literature, graphic and pictorial methods of instruction are becoming available to refinery training departments. A number of universities and colleges, through their industrial training departments, have been making progress in the preparation of special courses. A number of such courses have been validated by the Committee on Vocational Training of the American Petroleum Institute. The immediate future will see much emphasis placed on the need for vocational training in refineries. Men with basic knowledge of the physical sciences are needed in every refinery operation. These men must know more about physics and chemistry and the practical application of these sciences to their work.[14,16] The future of refining will see such training efforts developed to a high degree, and good employer-employe relations will be made even better by the active participation and cooperation of labor groups.

The petroleum industry has been particularly favored by much better than average employer-employe relations from which have come top wages, good working conditions, retirement plans, group-insurance plans and other advantages enjoyed by employes of very few other industries. The people employed in tomorrow's refineries will see that these advantages are retained. With a spirit of willingness on the part of both management and labor to sit down together and solve the human engineering problems created by a great and growing industry, mutual benefits will be gained for both, and America will enjoy an even higher standard of living than it does at this time.

Conclusions

What do these prospects of the refinery of the future mean to the capable and interested refinery employe?

They mean that he may enjoy a sense of security as regards continued employment in his field of work; that there is a growing demand for the products of his labor; and that research is pointing out new methods and new techniques which will make his work less burdensome and with which he must become familiar. They mean that for him there is almost unlimited opportunity to improve his usefulness to his organization; that his reward will be in proportion to his ability to produce; and that through cooperation of management and labor, mutual benefits are certain for all concerned. They mean that he may face the challenge of tomorrow with faith, hope and enthusiasm.

Acknowledgment

The author is indebted to E. E. McPherson and J. P. Langfitt of The Pure Oil Company for permission to publish this series of articles. He also is indebted to Dr. P. S. Nisson and J. R. Harrison for assistance in the preparation of the manuscripts.

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Miscellaneous Fractionating Techniques

THE fractionation of hydrocarbon mixtures in standard bubble-cap plate towers is discussed in a manner designed to emphasize the capacity and efficiency factors for existing equipment. A brief theoretical discussion is followed by sections on operating procedure, instrumentation, changes outside towers, and changes inside towers, to familiarize operators with certain techniques used by the authors to improve the performance of fractionators. An engineering investigation of a specific tower, covering the development of improved performance, is appended as an illustration of the application of parts of the text of the paper. The authors collaborated during the recent war to prepare the paper so that units of the industry might better be able to maximize war-products outputs.

This paper was presented to a Group Session of the Division of Refining on Gasoline before the Twenty-sixth Annual Meeting of the American Petroleum Institute, Chicago, November 12, 1946.

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DURING the war the petroleum industry, through the Petroleum Industry War Council, gave freely of technical knowledge and manpower that the armed services might not lack petroleum products. This service was spearheaded by the Technical Advisory Committee (a subcommittee of the Technical Committee of the council) under the chairmanship of T. G. Delbridge of The Atlantic Refining Company. With K. G. Mackenzie of The Texas Company and A. E. Miller of Sinclair Refining Company as its secretaries, this group collected from the entire industry a great deal of valuable technical information which was distributed, at the direction of the Petroleum Administration for War, to all those able to use it in advancing our success in the war.

One of the assignments of TAC Primary Processes Subcommittee was to develop all possible information on how to get the most in capacity and efficiency from bottleneck fractionation equipment. This study was one of a number of TAC reports on fractionation, and is presented here because of its continuing postwar interest and its bearing on future fractionating tower designs and operations.

The authors wish to take this opportunity to thank and credit not only their own companies, but the many others whose findings are reflected in this paper.

Explanatory

This report has been prepared in order to present to the petroleum industry some of the generally applicable techniques which have been utilized by various companies for eliminating obstructions to light-ends tower capacity or efficiency.

It is felt that the ideas and principles included may prove beneficial in aiding other refiners with similar problems to increase their tower capacities or improve the quality of their products.

This report is not intended as a textbook on the theory of fractionation nor as an instruction book on the operation of fractionators. However, it was felt that, inasmuch as this report will undoubtedly be read by non-technical men in charge of operations, a short theoretical description of fractionation would be helpful in clarifying some of the statements and comments contained in the body of the report.

The body of the report contains a description of possible tower bottlenecks and some reasons for poor efficiencies. The items discussed are by no means all that are possible, but include those which have been met with by the authors. The description covering these items is kept necessarily short and incomplete for sake of conciseness, inasmuch as the actual conditions in any one case are complicated and varied. It is hoped that these short comments on various bottlenecks and sources of poor efficiencies will stimulate thought along similar lines, so that the individual problems can be more readily solved and the fractionation improved.

No attempt is made in most cases to segregate those changes which would improve fractionation from the changes to increase capacity. Usually if efficiency is improved, increased capacity is attainable when separating at the original efficiency. Similarly, removing bottlenecks in the fractionating equipment proper usually will improve efficiency if the loads are not increased. In most cases, both are desirable. Items such as mechanical difficulties in equipment adjacent to the fractionator tower and recommendation of correct instrumentation were considered to be beyond the scope of the report. Packed and perforated plate towers were not considered.

A detailed report covering test data and calculations covering a fractionating tower that was operating poorly is included to illustrate a method of attack when trying to improve the operation. The calculation methods herein presented are not to be taken as recommended methods, but are given as an example of what should be calculated.

What is a Fractionating Tower?

In order to understand the function of a fractionating column and to correlate this understanding with the discussions that follow, two concepts of the column will be given. The first, although not rigorous scientifically, will permit a visualization of the operation. The second is theoretically sound, but possibly not so easily visualized.

The first concept is to consider the fractionating column as two separate and distinct units. The bottom, or stripping unit, will remove from the feed all constituents not desired in the bottoms product. The stripping of the feed in this section of the column is accomplished by a stream of vapor originating in the reboiler of the column. This vapor passes up through the trays, and strips from the downflowing liquid the constituents that are desired in the overhead from the column. Constituents desired in the bottoms also are partly stripped from the descending liquid, so that from the top tray of this stripping section all the overhead constituents

* Now with Fish Engineering Corporation, Houston, Texas.

and some of the bottoms constituents will be found in the vapor phase.

These mixed vapors pass up into the fractionating or "absorbing section." The top part of the column can be visualized to consist of an absorber wherein the constituents desired in the bottoms are absorbed by the reflux. In the ordinary absorber the reflux (or lean oil) is relatively non-volatile, and passes out of the bottom of the absorber together with the material that has been absorbed from the rising

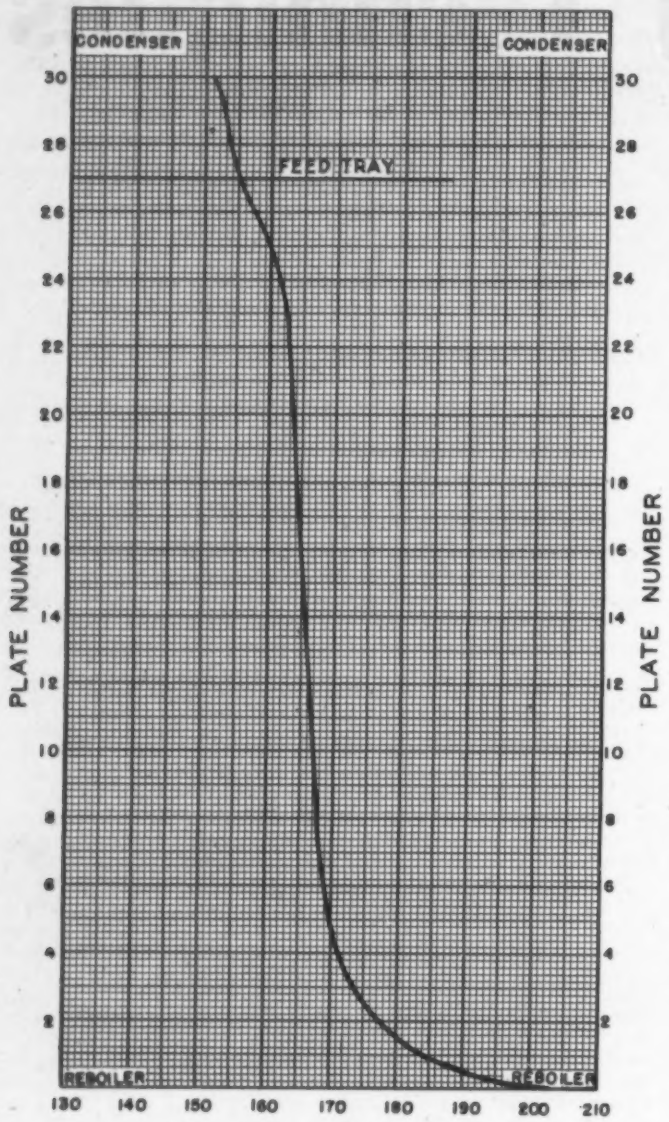


FIGURE 1

Calculated Temperature Gradient—Primary Debutanizer

vapors. In a fractionating column, the reflux is a relatively highly volatile liquid that is vaporized on passing down through the column, thereby condensing and concentrating the constituents desired in the bottoms, returning them to the stripping section.

From this concept, it follows that there is a minimum quantity of vapor required to pass up through the stripping section in order to remove the undesired constituents completely from the bottoms. Likewise, in the fractionating (or absorbing) section there is a minimum quantity of liquid that will absorb the bottoms constituents (that have been carried above the feed tray by the vapors) from the rising vapors and return them to the stripping section.

The second concept is to consider a fractionating column as a heat engine. For any given degree of fractionation there is a minimum amount of energy required to separate the overhead product from the bottoms product. The heat input into this heat engine is supplied by the feed and the reboiler. The heat that is introduced into the column is removed by the reflux and finished products.

Concentration and Temperature Gradients

In fractionating columns the temperature gradient is not usually a straight line nor a uniform curve. Likewise, the concentration gradient of any component may change materially from point to point in the column. Figures 1, 3, and 5 show typical temperature gradients in fractionating columns. Figures 2, 4, and 6 are concentration curves corresponding to the aforementioned temperature-gradient curves.

Temperature gradient (Figure 1) and composition (Figure 2) are typical of a column that is used to strip from the bottom product a large percentage of the lighter components, but is not producing an overhead product with a high percentage of purity. As this job is primarily one of stripping, the feed tray will be located relatively high in the column. Referring to Figure 2, it will be noted that the two heaviest components in the overhead (except a small amount of pentane), viz., butane and butylene-2, show a point of maximum concentration approximately four trays above the bottom of the column. This point of high concentration, with rapid decrease below that point, is usually associated with a rapid change in temperature from tray to tray below that point. Above that point, there is usually a section of the column where the temperature change from tray to tray is only slight. However, in this section of almost constant temperature, a rapid change in composition may still take place. Figure 2 shows that, while a rapid change of composition of *iso*butane is taking place in this section, the other components are changing their composition relatively slowly. In this column, the most sensitive control point would be on the 24th or 25th tray from the bottom where the temperature gradient is changing rapidly. This point is discussed more fully later in the report.

Figures 3 and 4 show temperature and concentration gradients in a typical depropanizer operating to strip the bottoms of propane with a small loss of butane overhead. Again the main job is one of stripping, so the feed-tray location is relatively high in the column. The temperature gradient in this column has the same general characteristics as the temperature gradient of Figure 1, except that a much larger temperature gradient exists in the top part of the column.

Figures 5 and 6 show the composition gradients and temperature gradient of a typical de*iso*butanizer operating in an alkylation plant. The temperature gradient and composition gradient are similar to those previously shown. It is interesting to note in Figures 5 and 6 that the temperature gradient in the stripping section shows the highest slope between trays 30 and 40. Over this section of the column, it will be noted that the greatest reduction in *iso*butane content takes place (Figure 6). This would be a sensitive point of control. Above the feed, the sharp change in temperature between feed plate and tray No. 60 is due primarily to fractionating out the alkylate vaporized from the feed. The rate of change in temperature decreases over the next few trays, and then increases again where the normal-butane concentration of the overhead decreases, and the propane concentration increases rapidly. The other point of temperature control, to be most sensitive, would be near the 66th or 67th tray. It must be realized that other compositions and other degrees of cut will produce temperature gradients and composition gradients somewhat different from those shown, but the general characteristics in all cases will be the same.

Fluid Flow on Bubble-Cap Trays

a. *Flow rates:* There are two distinct types of fluids with entirely different flow paths flowing in a fractionating tower. Gas or vapor, the major quantity of which usually originates in the reboiler, flows up the column. In theoretical fractionation calculations, it is usually assumed that the mols of vapor in all sections of a tower are the same when the charge enters at its bubble point. In actual towers, this assumption is only approximated, and in unusual cases the quantity of vapor varies considerably. If the feed is partly vaporized, this vapor combines with the vapor flowing up from the bottom of the tower and, hence, the quantity of vapor just above the feed tray is larger than that just below the feed tray. If the charge comes into the tower below its bubble point, additional vapor must be present in the section below the feed to heat the charge to its boiling point. Because the vapor from a reboiler frequently has a much higher temperature than the bottom tray, this vapor contains more heat in relation to its volume than vapor passing through the second tray from the bottom. Hence, the volume of vapor passing the first few bottom trays may be larger than the vapor from the reboiler.

Liquid (the other type of fluid) is introduced at the top of the tower as reflux, and flows down the tower, crossing each

tray. At the feed tray, this internal reflux is combined with the charge liquid and continues down the tower. Normally, the quantity of liquid in the bottom section of a tower is larger than the quantity in the upper section. However, if the charge enters substantially all vaporized but not superheated (at its dew point), the quantity of reflux liquid is about the same in any section of the tower. When the charge comes in above its dew point (superheated), additional cooling in the form of reflux to the feed tray from the top section must cool the charge to its dew point; hence, the liquid overflow in the top section will be larger than in the bottom section. Liquid side-stream drawoffs will decrease the quantity of liquid overflow below the point of drawoff. If reflux is introduced into a tower cold or well below its bubble point, the reflux quantity will increase until it reaches its boiling point by cooling and condensing some of the vapors.

b. *Vapor path:* In passing from tray to tray the vapor goes through the uptakes, reverses its path, and flows in the opposite direction in the annular space between the caps and the uptakes. It changes direction again to go through the slots, and finally bubbles up through the liquid on the tray.

The pressure drop between the trays is the sum of the pressure drop through the uptakes, reversal section, annular section, slots, depth of equivalent liquid at the point of issuance of the vapor from the slots, and various entrance and exit losses. The following concept of vapor bubbling through liquid is idealized for sake of clarity.

At low slot velocities, the vapors form large bubbles in passing through the liquid. Under these conditions, only a small part of the liquid comes in contact with the vapor, and the tray efficiency will be low. At moderate slot velocities, the bubbles will be smaller and the liquid will be well agitated, and a high tray efficiency results. At high slot velocities, the bubbles break down and coalesce with one another, and vapor channeling results. This results in a lowering of tray efficiency, as the vapor does not come in good contact with liquid.

Visual observation of bubbling trays shows that, at moderate and high gas velocities, the liquid is well agitated and is broken down into foam or large droplets which bounce across the tray. These droplets occupy a zone that is higher above the top of the tray than that which would be occupied by clear liquid if no vapor were passing through the liquid. Above this zone there occurs another dense zone of smaller droplets which extends 12 to 24 inches above the tray. The height of the second zone depends on the slot velocities, superficial velocities, liquid depth and rate, and increases with increase in superficial velocity. It is this zone that should be kept in the space between the trays; for, if it extended higher than the space between the trays, serious decrease in tray efficiency would result due to entrainment. Above this zone there is another zone composed of a fine mist that extends for a considerable distance above the middle zone, and will always extend to the next plate. The percentage entrainment

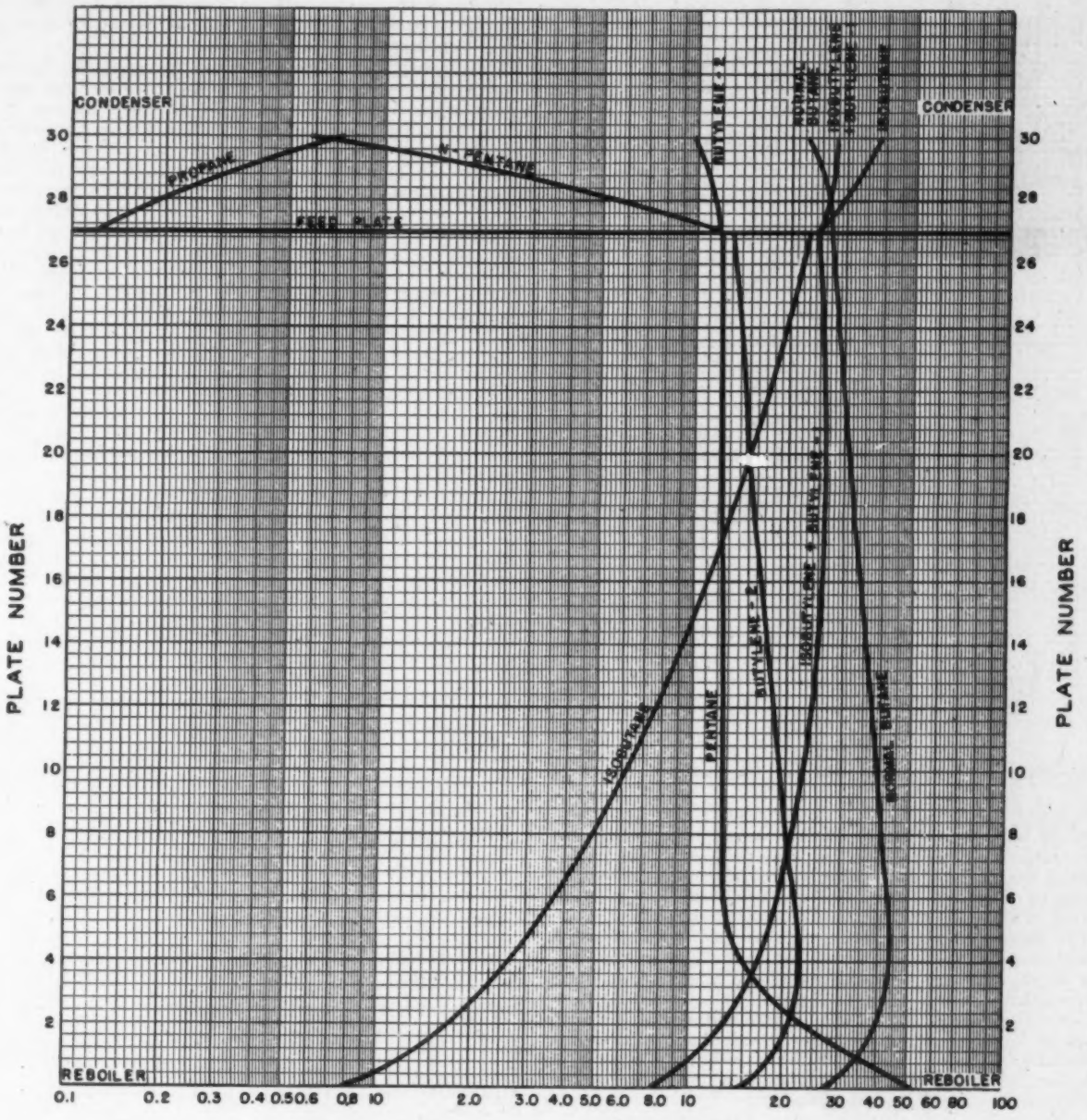


FIGURE 2
Plate Liquid Composition—Primary Debutanizer

that this mist represents is small and, hence, has only a small effect on tray efficiency. There is one other zone that sometimes can be observed. This is one composed of clear, unagitated liquid and lying next to the plate surface, underneath the slots, and in any dead spaces in the liquid path across the tray. If this zone is large, the tray efficiency is low, as it represents liquid that is not in equilibrium with the vapor.

c. *Liquid path:* The liquid flows across the tray between and

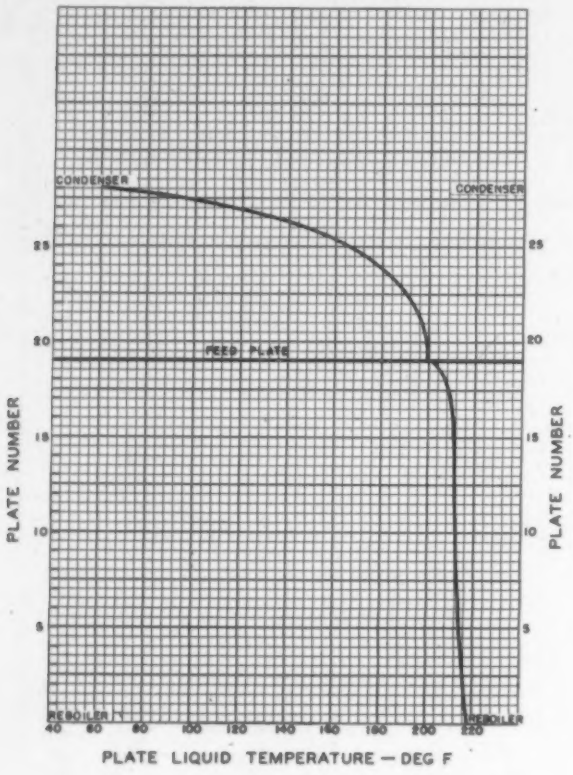


FIGURE 3
Calculated Temperature Gradient—Depropanizer

over the caps, and will take the path of least resistance. In trays which have bubble caps located close together, there is considerable resistance to flow, and the depth of liquid on the inlet side will be considerably greater than that on the outlet side of the tray. Under this condition the majority of the vapors will pass through the caps on the outlet side where the liquid depth is low, and may not bubble at all from caps near the inlet side. If the hydraulic gradient is sufficiently large, the caps near the inlet side may allow liquid to backtrap through the risers to the tray below. If the vapor is not well distributed through all caps and risers, serious decreases in the tray efficiencies may result due to excessive slot velocities at some points. Another effect in crossflow trays is that the vapor will flow across the top of the trays countercurrent to the liquid flow in order to pass through only the caps near the overflow weirs. This crossflowing of the vapor causes an added piling up of liquid at the inlet side of the tray, and thereby accentuates backtrapping of liquid through the uptakes. Whenever backtrapping occurs, serious decrease in tray efficiency results.

At the outlet side of a tray the liquid flows over the weir into the downflow. In modern towers the pressure drop through the downflow is negligible except at the constriction where the liquid leaves the downflow and enters the tray below. The function of the downflow area is to provide space to disengage vapor that is carried over the weir with the liquid. A dead space of liquid just before the weir also accomplishes vapor disengagement. The vapor must be disengaged from the liquid, or the liquid-carrying capacity of the downflow will be markedly decreased. The height of liquid standing in the downflow must be that which counterbalances the pressure drop between the trays plus the liquid gradient plus the pressure drop through the downflow. When this liquid level reaches the top of the weir, the tower will begin to flood. In cases where vapor is carried through the downflow, the apparent density of the liquid is decreased and the liquid height raised.

A row of bubble caps normal to the direction of flow of liquid across the tray will stop bubbling when the magnitude of the liquid gradient at this point equals the average pressure drop through the caps bubbling. A row of caps will begin to backtrap when the liquid gradient at this point equals the average gas-pressure drop through the bubbling caps plus the distance between the top of the slots and the top of the uptakes. Thus, it can be seen that poor vapor distribution through the caps will result when the pressure drop through the caps is low, even though the liquid gradient is also low. A tray will be unstable, with the danger of backtrapping ever present, when the pressure drop through the cap is low and the distance between the top of the slots and top of the uptakes is also small.

d. *Changes in flow rates:* In the foregoing discussion it has been assumed that steady rates of flow for both liquid and vapor were present. However, in actual practice both are continually fluctuating. An accelerating flow produces an additional pressure-drop quantity due to the acceleration, whereas a decelerating flow produces a negative pressure-drop quantity. A rapid acceleration of charge or reflux causes a high temporary liquid gradient that either causes the plate to back-trap or to flood. A rapid acceleration of vapor-flow rate pushes the liquid away from the caps and blows the liquid off the trays. A rapid deceleration of vapor flow produces a negative pressure drop through the caps, and will allow liquid to backtrap through the caps that are more highly submerged. The rate of acceleration or deceleration is more important in producing these effects than the absolute value of liquid rate of vapor rate experienced during the change. All these effects, of course, cause poor efficiency; and, if the changes in rates occur continuously, a certain percentage of the plates in the tower will be upset at any one time, and the tower will exhibit a poor average efficiency. At low loads, the vapor rate through the caps has a tendency to surge, thereby causing backtrapping and low efficiencies. The liquid distribution is also poor at low loads.

e. *Overload conditions:* Overload conditions are present where vapor and liquid rates are such that a "flooding condition" exists. A tray will begin to flood when the liquid level standing in the downflow from the tray reaches the top of the overflow weir (see Figure 17). A slight increase in load will then build up liquid on the tray until liquid fills the vapor space and enters the risers above. One section of a tower

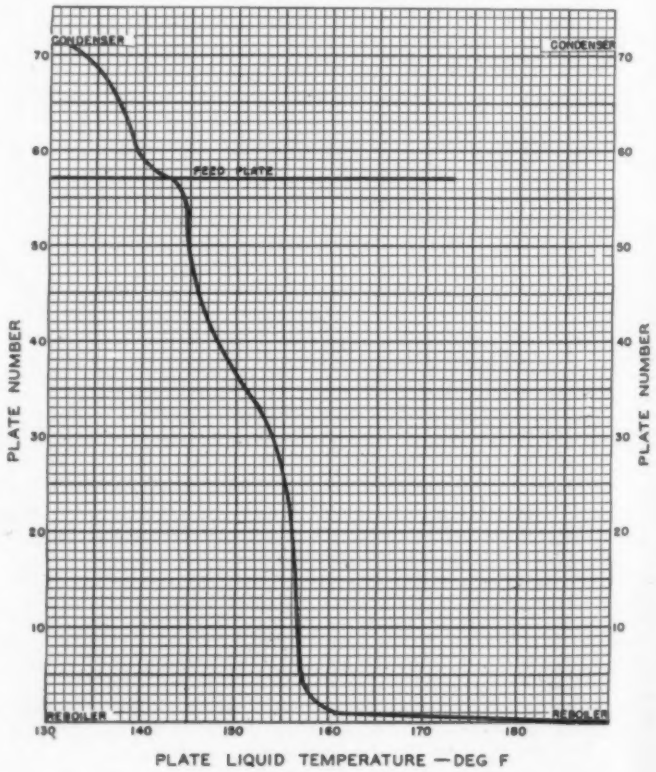


FIGURE 5
Calculated Temperature Gradient—Deisobutanizer

may flood while the balance of the tower can be below the flood point. Usually flooding begins on the feed tray. When a tower floods in the top section because of flooding below the feed, liquid may be carried over in the vapor line.

When flooding occurs, there is usually a break in the pressure gradient through the tower at the region of flooding with

a much higher gradient throughout that region. *When liquid is being carried overhead, additional pump-back reflux will have no effect on the top temperature or reboiler load.* This is due to the fact that a flooding downflow will handle only a fixed amount of overflow liquid, which is usually less than the non-flooding downflow capacity. Additional pump-back reflux over this amount will be carried overhead directly as a liquid, and will never enter the section of the tower below the top tray. By reboiler fills up to the bottom tray with liquid due to fouling of the reboiler. This flooding of the bottom downflow will cause the balance of the tower to flood even though the tower has ample capacity.

Liquid being carried overhead is not a true indication of flooding, as this carry-over might be due to entrainment only. However, entrainment alone does not cause large amounts of liquid to be carried over.

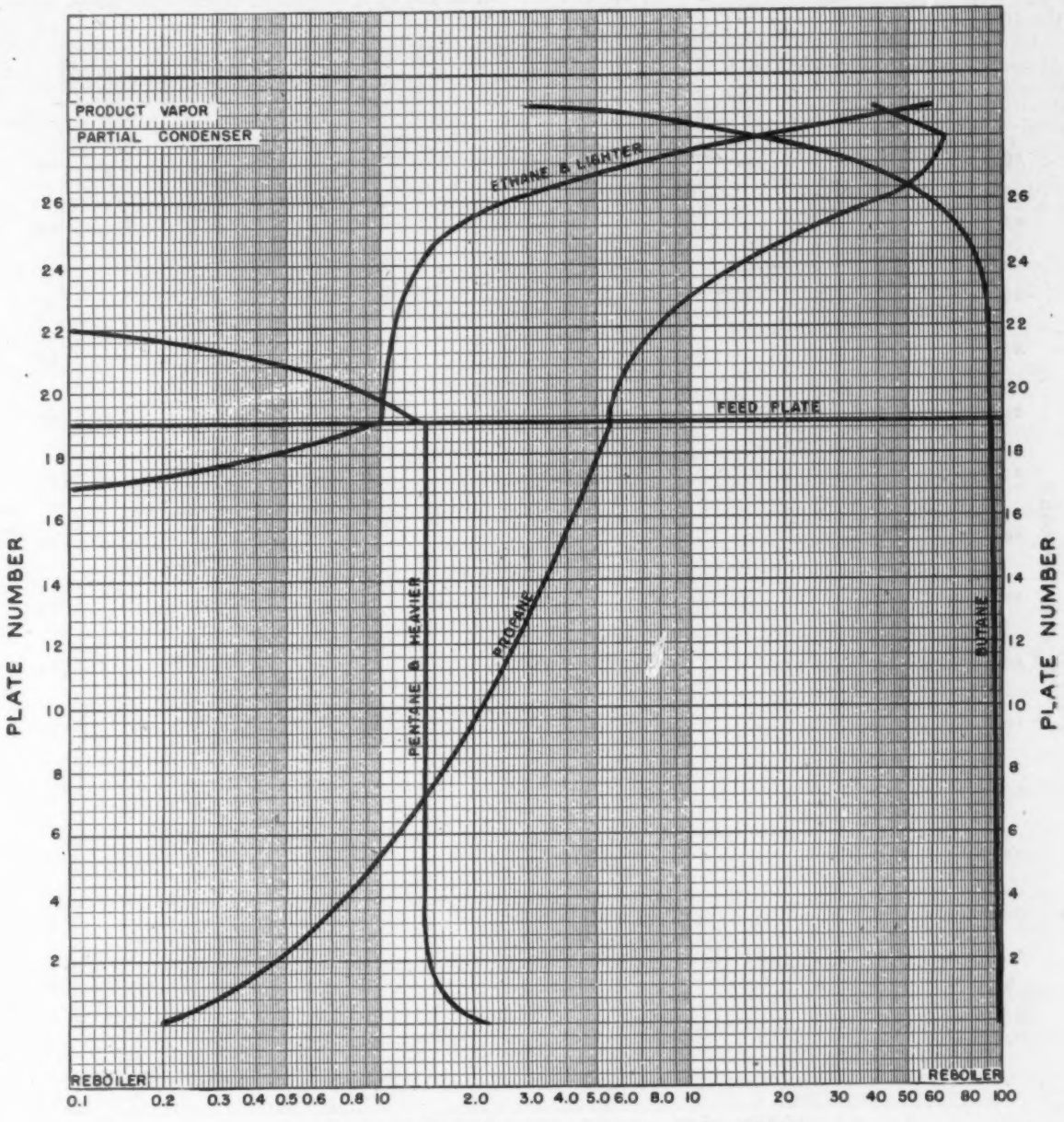

PRODUCT VAPOR
PARTIAL CONDENSER
ETHANE & LIGHTER
FEED PLATE
PENTANE & HEAVIER
PROPANE
BUTANE
REBOILER
REBOILER
PLATE NUMBER
PLATE NUMBER
LIQUID COMPOSITION — MOL PER CENT
0.1 0.2 0.3 0.4 0.5 0.6 0.8 1.0 2.0 3.0 4.0 5.0 6.0 8.0 10 20 30 40 50 60 80 100
2 4 6 8 10 12 14 16 18 20 22 24 26

FIGURE 4

Plate Liquid Composition—Depropanizer

making a heat balance over the tower or condenser, it can be shown that a portion of the stream entering the condenser is not in the vapor state. Inasmuch as the overflow liquid is limited, the reboiler load may not be so high as when flooding is not occurring. If the flood occurs at the feed tray, the bottoms-product quantity may be reduced temporarily.

As a rule when flooding occurs, poor fractionation results. This may be due either to slugging of liquid overhead ("puking," with resulting pressure and temperature variations), or to the increased tower pressure. In the latter case, the alpha values are decreased and the required separation is theoretically more difficult. Some towers have been observed, however, that have good efficiencies when the tower is operated in a flooding condition at the top of the tower only, but under these conditions there are no variations in temperatures or pressures. If flooding starts at the feed tray and works up the tower, poor separations may result.

Flooding will start when a totally enclosed trap pan to a

Purpose of Trays and Tray Efficiencies

a. *Trays:* The modern fractionating column usually consists of a number of trays having bubble caps so arranged that liquid flows across the trays. The vapor through the caps bubbles through the liquid on the trays. In this manner, intimate contact over a large surface area between the rising vapors and descending liquid is accomplished. Through this surface there is a material exchange between the liquid and vapor, the exchange occurring in both directions simultaneously. At the same time material is exchanged, heat is also exchanged. Because the vapors are at their dew points and liquids at their bubble points on the trays, substantially all the heat transferred on any tray is due to an exchange of latent heat. Therefore, the efficiency of material and heat transfer must be comparable.

b. *Efficiencies:* There are a number of different definitions of tray efficiency. For the purpose of this report, tray efficiency is defined as the ratio of the number of theoretical trays re-

quired for a given separation at a given reflux ratio and the actual number of operating trays required for that separation with that reflux rate. A theoretical tray is one on which the liquid leaving the tray is in thermal and material equilibrium with the vapor leaving the tray. The two main factors that may affect tray efficiency to a large degree are underload and overload.

If, at any given point, the liquid is considered as flowing across the tray and vapor rising through the tray, there will practice, however, the column usually is equipped with more than the calculated number of actual trays.

The additional trays are for the purpose of compensating the effects of the many operating variables such as feed rate, feed composition, degree of vapor-liquid contact, inaccuracies of control caused by instrument lag, and fluctuations in utility quantities or temperatures. The number of trays over the actual calculated required will largely depend on the magnitude and rate of change of these variables.

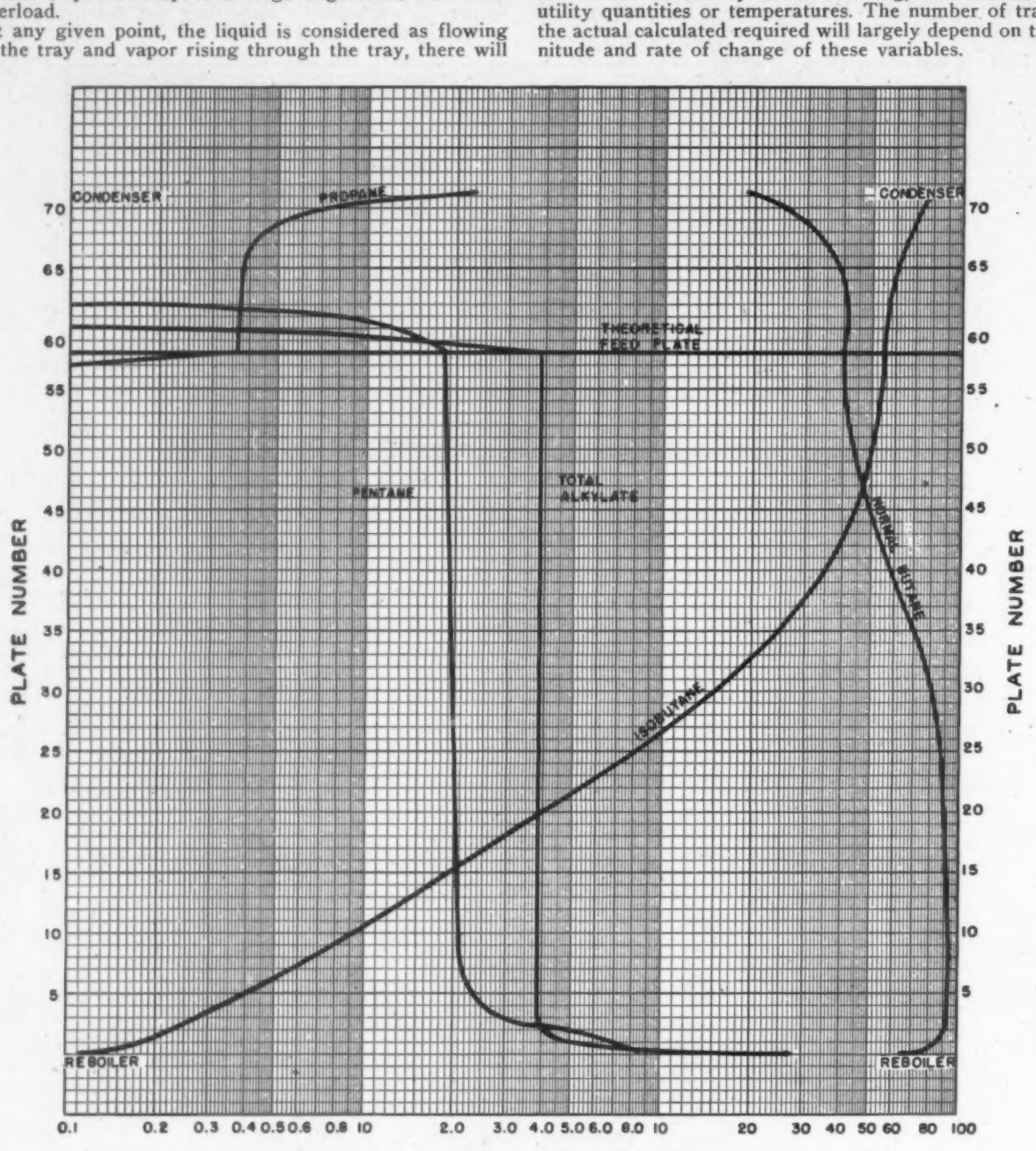


FIGURE 6

Plate Liquid Composition—Deisobutanizer

be a certain ratio between the equilibrium actually obtained and the theoretical equilibrium that could be obtained if sufficient time and surface were allowed. The efficiency at this point is termed "point efficiency."

On a crossflow tray, where the liquid is changing in composition from the inlet to the outlet side and where the vapor entering the tray has substantially a constant composition, the summation of these point efficiencies may be considerably different from the overall plate efficiency. In fact, in a number of cases where point efficiency is high, the overall plate efficiency may actually exceed 100 percent.

c. *Number of trays:* For any given separation of a given feed there will be a certain definite fixed number of theoretical equilibrium steps that must be passed through in order to achieve the desired result with a given reflux rate. The actual number of trays required for the separation is then the theoretical steps divided by the overall tray efficiency. In

Discussion of Reflux

a. *Purpose of reflux:* Reflux is introduced into a column to prevent constituents that are desired in the bottoms product from passing overhead. This reflux, in passing down through the column, absorbs the heavy constituents and by latent heat exchange the reflux is partly vaporized. The unvaporized reflux from the fractionating section, together with the absorbed (condensed) constituents that are desired in the bottom product, flow into the stripping section. The liquid constituents that flow from the fractionating section into the stripping section furnish most of the vapors that are used in stripping the overhead product from the bottoms. The total vapor is made up of vaporized reflux plus the lighter constituents of the feed. When producing a bottoms product of constant composition, additional heat in the reboiler will not affect the temperature of the bottoms product.

b. *Definition of reflux ratio:* The reflux to a column is de-

fined normally as a ratio rather than as an absolute quantity. For specific purposes, this ratio is taken at different points in the system. Various expressions of reflux ratio in common use are the ratio of reflux to feed, the ratio of reflux to bottoms, the ratio of the liquid flowing down inside the column to the vapor rising in the column. In this last case, there are two internal reflux ratios, viz., one above the feed and the other below the feed.

Still another reflux ratio (as used principally throughout this report) is the ratio of the reflux from the top tray to the net overhead product. For engineering purposes, it is customary to express the reflux ratio on a mole basis.

c. *Infinite reflux ratio:* There is a hypothetical case, used principally in calculations, in which a column is considered to be completely shut in; i.e., no feed entering the column, no overhead products or bottoms products being withdrawn. Under these conditions, the total overhead is condensed and returned to the column as reflux, and the reflux ratio becomes infinite. In this case, the absolute minimum number of theoretical trays or equilibrium steps is required to accomplish a given separation.

d. *Minimum reflux:* Considering the column as a heat engine, there is a minimum amount of heat required to make any separation; and it follows, therefore, that, irrespective of the number of trays in the column, there is a minimum reflux required for any degree of fractionation.

e. *Actual reflux:* It has been found that the actual reflux required in a properly designed and operated fractionating unit is not far greater than the minimum reflux ratio. It is usually most economical to operate a column with the actual reflux ratio from 1.2 to 1.4 times the minimum. If the column is operating with reflux ratios substantially above these limits, increased reflux will make no appreciable improvement in fractionation. *The reflux ratio for a given purity of overhead and bottoms products will vary roughly, inversely with percent of overhead product in the feed.* Consequently, the reflux for such a separation bears an almost constant ratio to the feed rate.

Feed

a. *Feed-tray location:* If the column is considered to consist of two parts, viz., the stripping section and the absorbing or fractionating section, it will be appreciated that, by varying the point at which the feed is introduced into the column, the number of the trays in the stripping section can be increased at the expense of the trays in the fractionating section, or vice versa. In many cases the stripping problem is more difficult than the fractionating problem, indicating that the stripping section should contain the greater number of trays. If the feed is introduced at too low a point in the column, insufficient trays are available for stripping, and there are more than enough trays for fractionation. Under such conditions, the apparent tray efficiency in the fractionating section will be very low. If, on the other hand, the stripping job is very simple and fractionation is difficult, the feed should be introduced relatively low in the column so that there are few stripping plates and a large number of fractionating plates.

b. *Condition of Feed:* One of the important factors for maximum column efficiency is the percent of feed vaporized at the point of feed introduction into the column.

Whether the job is one of easy fractionation or one of easy stripping, very often depends on the percent of feed vaporized before it enters the column. If a high percent of the feed is vaporized, the fractionating job becomes more difficult and the stripping job easier. Therefore, the point of feed introduction should be lowered as the percent vaporization of the feed is increased. It is considered normally good practice to *hold the percent vaporization of the feed down to the percent of feed taken overhead as product.*

For a given separation, with nominal feed vaporization, additional heat input to the feed will relieve an equivalent amount of heat from the reboiler. In a column overloaded below the feed and underloaded above the feed, a better distribution of load, together with higher fractionating efficiency or higher throughput, can be obtained by putting additional heat into the feed.

By the term "load" we mean the amount of liquid and vapor passing through the column. As an example of the effect of feed vaporization on the amount of reflux and load in the different sections of the column, reference is made to Figures 7 and 8. These show the reflux ratio, relative vapor rates, relative liquid rates, and heat loads applied as a function of percent vaporization of the feed. The study was made at constant feed rate, and with constant fractionation. The optimum feed point was assumed for all cases.

For the particular example, these show that as the percentage vaporization of the feed increases:

1. The required reflux rate rises slowly until a feed vaporization of 12 to 13 percent is reached, beyond which the required reflux increases more rapidly.
2. The liquid and vapor rates below the feed tray are sharply reduced for the first few percent of feed vaporized, the rate of reduction then tending to flatten out.
3. The liquid and vapor rates above the feed tray increase slowly for about the first 15 percent vaporized, beyond which they increase more rapidly.

It will be noted that the percent feed vaporized is exceedingly sensitive to temperature. Obviously, it would be difficult to control the percent feed vaporized by a temperature controller on the feed to such a column. Under these conditions, the type of feed heater should follow more or less the pattern of vaporizer to be described later on in this paper. The magnitude of these variations will vary with the feed composition and separation desired, but will tend to follow the general form of these curves.

Figure 8 shows the relative heat loads on the system as a function of the feed vaporized. It will be noted that as the percent feed vaporization increases from 0 to approximately 12 or 13 percent:

1. There is substantially no increase in the load on the reflux condenser.
2. The load on the preheater increases materially; but
3. The load on the reboiler decreases an almost equivalent amount.
4. The total heat load on the system remains substantially constant.

As the percent vaporized increases above 1 to 13 percent:

1. The load on the reflux condenser increases.
2. The reboiler load remains substantially constant.
3. The preheater load increases.
4. The total heat load on the system increases.

Absorber Characteristics

An absorber is similiar in many respects to a fractionating column. However, in normal absorber operation, material exchange takes places in one direction only, i.e., from the gas phase to the liquid phase. The absorbing medium is usually of low volatility, so that the amount of absorbing medium vaporized is negligible. Because material exchange is primarily in one direction, entrainment of liquid from tray to tray is very detrimental to overall efficiency.

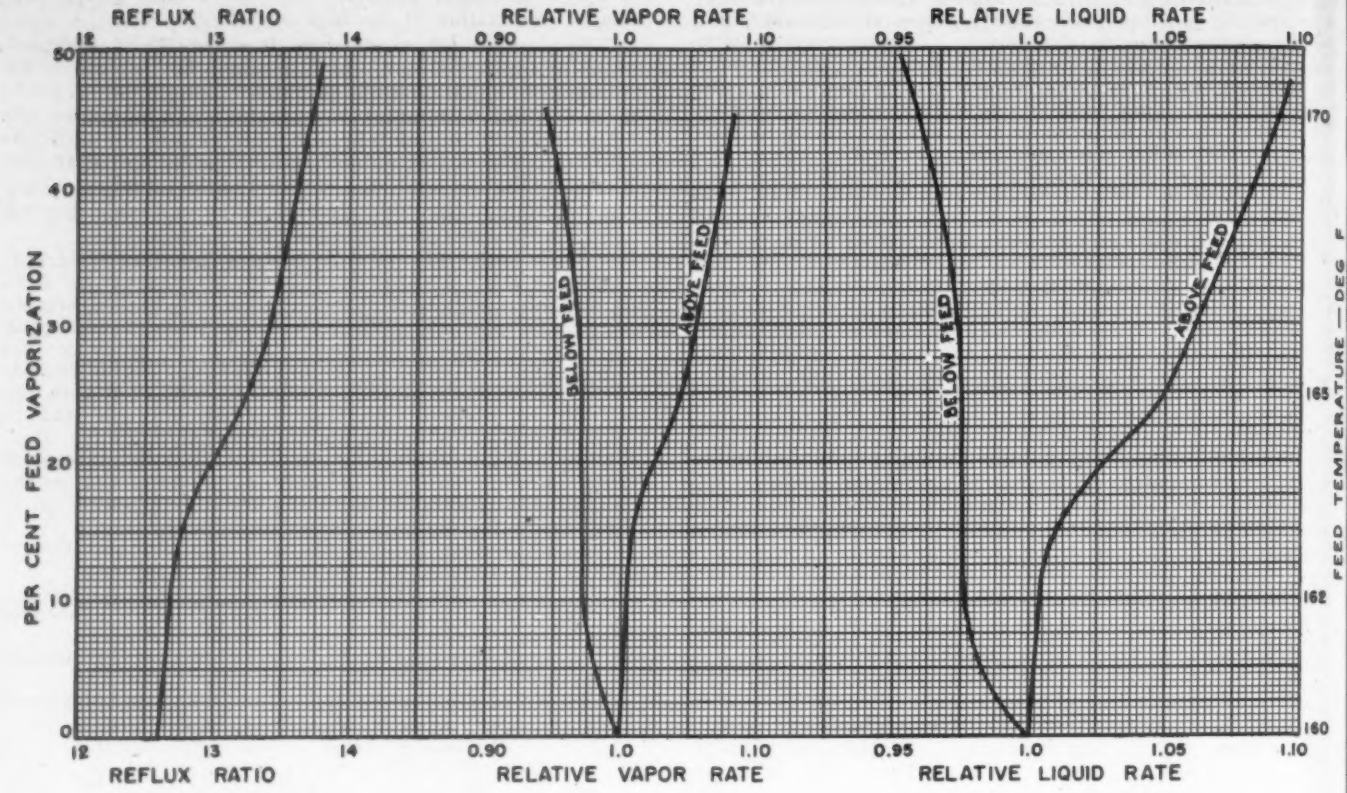
There are a number of conditions in absorber operation on very rich gas, especially at pressures in excess of about 100 psig in which the heat of absorption may be great enough to raise the temperature of the oil passing through the absorber sufficiently to distill considerable quantities of the lighter constituents from the oil near the bottom of the absorber. These constituents would be reabsorbed, in turn, in the upper part of the absorber—setting up a large internal recycle vapor load. This recycle would materially decrease the overall absorber efficiency.

The heat capacity of the gas entering the absorber may tend to drop the bottom-tray temperature of the absorber. A combination of the cooling effect of the incoming gas, together with vapor recycling within the absorber, would have the effect of producing in the center to upper part of the absorber the highest temperatures in the column. In order to combat this effect when operating on rich gas, it is customary to use intercoolers in order more nearly to approach a constant temperature throughout the column. The intercoolers should be placed at the point of maximum heat generation in the absorber, rather than at the point where maximum temperature would occur if intercoolers were not used. This point is frequently well below the center of the absorber, and in some cases may be only three or four trays from the bottom.

Heat-Load Considerations

1. Effect on Product Quality

a. *Relation between heat input and heat removal:* In a given tower, the quality of the overhead and bottom products, from a feed of given composition and rate, is a function of: 1, the product rates as percent of the feed; 2, liquid downflow rate in the fractionating section; 3, stripping vapor rate in the stripping section; and, 4, the feed point. Liquid downflow rate in the fractionating section is roughly proportional to the reflux duty. The stripping vapor in the stripping section is approximately proportional to the reboiler heat input. A common practice is to set the feed temperature (heat content) and reflux rate constant at values which will result in



COLUMN FEED MOL %		BOTTOMS	OVERHEAD
PROPANE	0.40		1.63
ISOBUTANE	24.00	0.40	96.34
N. BUTANE	37.50	49.05	2.03
N. PENTANE	38.10	50.55	
	100.00	100.00	100.00

OPERATING PRESSURE 85 P.S.I.G.
NUMBER OF TRAYS 60
OVERHEAD 24.6 % OF FEED

FIGURE 7

Effect of Feed Preheat—Vapor and Liquid Rates

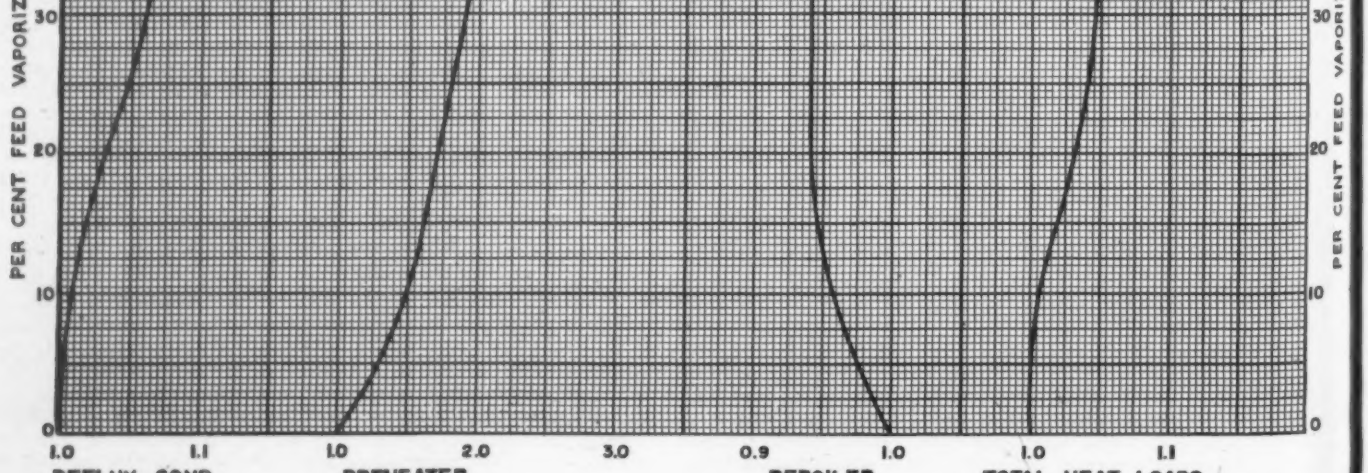


FIGURE 8

Effect of Feed Preheat—Relative Heat Loads

a reflux duty and a reboiler duty of the general order of magnitude required for the separation. The reboiler duty is then adjusted manually or automatically to maintain the exact difference between heat input and output required to maintain the one, and only one, rate of products corresponding to the desired quality. In some cases the reboiler duty is set constant and the reflux rate raised.

Although the reflux and reboiler duties usually can vary several percent without appreciably affecting product quality, the relation between heat input and heat output in Btu per hour, as expressed by heat content of feed plus reboiler duty minus reflux duty, must be maintained at an exact and precise amount in order not to impair seriously the quality of products. This is a very small fraction of 1 percent of the reflux duty or reboiler duty. The maintenance of this exceedingly precise relationship between heat input and heat output is the crux and essence of control.

In general, increasing the overhead product rate as percent of the feed increases the percent of high-boiling impurity in the overhead product, and decreases the percent of low-boiling impurity in the bottoms product.

All of the foregoing is illustrated by the case of a debutanizer taking a butane-product overhead. The reflux rate is set for constant rate, and the feed temperature and composition are constant. With reboiler duty at a given value, the butane product contains 1 percent of C_5, and the bottoms 0.03 percent C_4. Assume now that the reboiler duty is increased slightly to increase the overhead rate about 1 percent without changing the reflux rate. As the tower is already taking out all but a trace of C_4 from the bottoms, the 1 percent increase in overhead rate must of necessity consist almost entirely of C_5, which increases the C_5 in the total overhead to 2 percent, while the C_4 in the bottoms will drop from its previous value of 0.03 percent to, say, 0.02 percent. Routine analytical methods could hardly detect the change of C_4 in the bottoms, and it would probably be reported in both cases as 0 percent, or a trace. Assuming that the reflux rate was six times the overhead-product rate, the change of 1 percent in the latter corresponded to a change of 1 percent of 1/7=0.14 percent of the total vapor leaving the top of the tower, and a Btu-per-hour increase in condenser duty of 0.14 percent. The increase in reboiler duty which caused this change in product quality was the same small amount in Btu per hour as the increase in condenser duty. However, if the reflux rate had been increased to maintain the same difference between heat input and output, there would have been no appreciable change in product compositions.

b. *Change of reflux rate and reboiler duty at constant feed heat content and feed point:* The heat removed by a given amount of pumped reflux depends upon its temperature in relation to tower-top temperature. In flooded condenser type of control, different degrees of subcooling can change the amount of heat which the reflux removes by 20 percent or more. This discussion is concerned primarily with the effect of variations in heat removed by reflux, a function of both rate and temperature of reflux. For convenience, it will be assumed that the reflux temperature is not changed, so that the heat removed by reflux is proportional to its rate.

For a feed of given composition and given heat content, the reflux rate and reboiler duty go up and down together. If the tower is in balance, and additional heat is put in at the reboiler, it must be removed by pumping more reflux; or if the tower is in balance, and more reflux is pumped (removing more heat), the reboiler must supply the additional heat.

The quality of both products from feed of given rate, composition, and heat content will be increased for a given tower within limits fixed by the number of trays and by the maximum load which the tower can carry efficiently, by increasing reflux rate (and corresponding reboiler duty). There is a point above which the additional benefit will not compensate for the additional utilities consumed. Before this point is reached, it is possible that flooding or a falling off in fractionating efficiency may occur as a result of overload on one or both sections of the tower.

Maximum feed capacity for products of given quality calls for maximum reflux rate (and reboiler duty) consistent without overloading either section of the tower to flooding point or to the point where efficiency begins to be impaired seriously. Feed rate can be still further increased if a sacrifice is made on the quality of one or both products.

In cases where reflux is limited by condenser surface, fouling, or cooling-water temperature, and where there is surplus reboiler capacity, additional reflux can be obtained by raising the tower pressure, provided the allowable mechanical design pressure of equipment is not exceeded. The increase in tower pressure actually will require somewhat more reboiler duty and reflux to make the same separation, but this may be more than compensated for by the increased reflux available at the higher pressure.

In cases where the capacity of the reflux pump limits the amount of reflux, additional effective reflux inside the tower may be obtained by subcooling the reflux as much as possible. However, as pointed out elsewhere, such subcooling reduces the fractionating efficiency of the top tray. Hence, the net result of subcooling may be beneficial or it may be harmful.

c. *Warm reflux:* An improvement in the separation can sometimes be obtained by using reflux having a temperature approaching the top-tray temperature rather than by using a reflux well below its bubble point. In changing from a cold pump-back reflux to a warm pump-back reflux, the quantity being pumped will have to be somewhat increased so that the total cooling duty on the tower is kept the same. The increased external reflux, of course, does not increase the internal reflux in the tower. Using cold reflux requires that some of the tray area be used to transfer heat to the reflux to bring it up to its boiling point. The area used for heating the reflux will give poor tray efficiencies, because the liquid on the trays will not be boiling. The net effect of using warm reflux is to increase the number of trays in the tower by the one or more that effect separation.

2. Controlling of Heat Loads

It has been demonstrated by actual operation and tests of a depropanizer operating with feed of fairly uniform rate and composition that, when the tower is entirely on hand control, any two of the three items affecting heat, viz., feed temperature, reflux rate, and reboiler duty, can be set constant, and the tower controlled by adjustments of the third item to produce desired product quality. So far as the net results were concerned, it was immaterial, without regard to sensitivity, which of the three possible combinations was used. The reason for this is that the changes required for control are insignificant compared to the total reflux duty or reboiler duty.

Whereas frequently in the past it has been common practice to use the tower-top temperature or the temperature of the bottoms product leaving the system as a control point for automatically regulating the reboiler heating-medium rate or reflux rate, or as a guide point for manual resetting between inspection of products, there is a growing tendency in close fractionation to use one of the tray temperatures for this purpose. Although the temperature of the bottoms product and the tower-top temperature (in the case of pumped reflux having the same composition as overhead) have the advantage of being a function of product composition only and are not affected by changes of reflux rate or reboiler duty (only insofar as changes in these items affect quality), they have the serious disadvantage, in close fractionation, that the permissible variations in desired quality of products are too small to reflect corresponding changes in temperature at the top or bottom of the tower. However, these small changes in product quality are reflected in measurable changes in tray temperature in a certain region in the fractionating section and in the stripping section. Consequently, if heat is controlled to maintain a reasonably uniform temperature in either of these two regions, the product quality will be maintained within narrow limits, provided two other things are not changed. These two tems are:

In the section above the feed—

Change in composition of feed which results in change in proportion of light components in the overhead product.

Change in ratio of rate of liquid downflow to rate of overhead product.

In the section below the feed—

Change in composition of feed which results in change of proportion of heavy components in the bottoms product.

Change in ratio of rate stripping vapor to rate of bottoms product.

On account of the possibility of such changes, it may be necessary to hold a different temperature at different times at the point used for guide or control in order to make required product quality.

The choice of whether to use a tray temperature in the section of the tower above the feed or below the feed as a guide or control will depend, to some extent, on location of available thermo-wells. If suitable wells are available in both sections, it is considered more desirable to use that section from which a substantially pure product is taken off, or where there is least change in proportion of components in

the product other than the two between which separation is being made. The best region to use in a given section is the one in which there is the most change per tray in the percent of the component which, for the upper section, is the high-boiling impurity and, in the lower section, is the low-boiling impurity. It is not possible to generalize as to which tray temperature provides the best indication of product quality, except to say that it is probably in the middle third of the upper section or middle third of the lower section. This can be established by tray calculations or by experimental trial. On account of time lag, it may not be possible to locate a temperature controller at the optimum point as possible consistent with time lag, while the temperature at the optimum plant is watched as a guide for resetting the temperature controller.

In controlling a tower, adjustments are only made either manually or automatically by instruments, after indications appear that the control point has deviated from the norm. Then the operator or the instrument moves to correct the condition, and the tendency is to over-correct—tending to throw products off specification in the other direction. The net result is a weaving of product quality. In order to obviate over-correction, such changes should be made by a series of small increments of at least one-half-hour intervals. In most cases, longer intervals are necessary. Changes should be kept as small as possible to avoid flooding. The less frequently such corrections are required, the more uniform will be product quality; also, it will be possible to hold impurities in both products to smaller percentages. A tower can have many times the trays, reflux duty, and reboiler duty to make a given separation; but, if control is defective, one of the products will contain excessive impurities while the other product is purer than necessary.

Feed Consideration

1. Feed Rate and Composition

As a corollary of the foregoing, it follows that, for the maximum uniformity in quality of the two products, and also for maintaining a very small percent impurity in the two products, constant conditions that will not need frequent correction are required. It has been pointed out that a change in heat output or input in the order of a fraction of 1 percent will, if not corrected, result in an incorrect ratio of overhead to bottoms product, greatly increasing the impurity of one of the two products. In practice, such changes may occur as a result of changes in temperature of reflux, feed, or reboiler heating medium (if gasoil) even though all rates are constant. In addition, changes of feed composition which necessitate changes in ratio of overhead to bottoms product require heat adjustment, and may also affect tray temperatures used for controlling product purity. It has been found necessary, in some cases, to provide adequate surge capacity in the feed to a tower (particularly the first of a number of towers in series), in order to obtain reasonably constant feed rate and to smooth out variations in composition.

In a series of towers, removing propanes, then butanes, and then *iso*pentane from gasoline, it is necessary that the depropanizer maintain a reasonably constant ratio of C_3 to C_4 in the bottoms in order that tray temperatures used for guide or control in the upper section of the debutanizer will not be affected by a varying amount of C_3. Unless the debutanizer holds a very constant ratio of C_4 to *iso*-C_5 in the bottoms, the temperature in the upper section of the de*iso*pentanizer will be affected by varying quantities of C_4. As it is difficult to control the debutanizer in this way, the next best thing is to operate it for almost perfect debutanization by keeping a little C_5 in the overhead. In this way, there will be practically no C_4 in the *iso*pentane product of the de*iso*pentanizer.

2. Feed Point

There is an optimum feed point for obtaining, with the least amount of reboiler duty and reflux duty, the desired yield and quality of a product from a feed of given composition and percent vaporization. There is another, and perhaps different, optimum feed point for obtaining, with a given reboiler and reflux duty, the maximum yield of a product of specified quality. The optimum feed point varies with composition and percent vaporization of the feed. It is advisable to determine the optimum feed connection by actual trial of all those available, under normal operating conditions. If there is a major change in operation condition such as composition or vaporization of feed, or in ratio of reboiler duty or reflux to feed, it is advisable to reestablish the best feed connection for the new conditions by experimental trial. In some cases, changing feed connections will be beneficial. In other cases, it will make no noticeable difference.

When producing an overhead specification product such as *iso*butane or *iso*pentane of satisfactory quality but with substantial losses in the bottoms, it may be possible to increase the yield of product by merely raising the feed point. On the other hand, when producing a bottom specification product, such as butane, of satisfactory quality but with substantial loss in the overhead, it may be possible to increase the yield of product by merely lowering the feed point.

3. Heat Content of Feed

Usually the feed to a tower is partly vaporized, in which case increasing feed preheat results in increased vaporization, whereas a reduction of feed preheat results in decreased vaporization. The addition of heat is invariably accompanied by a temperature rise of the feed—although, as pointed out elsewhere, the rise may be so small in the case of a partly vaporized and extremely close-boiling feed that it is not a sensitive indication of heat content.

The effect of changing the heat content of a given feed cannot be separated from the resulting change of reboiler duty, or reflux rate. When the feed heat content is increased, the additional heat put into the system must be balanced out, either by pumping more reflux, by reducing the reboiler duty, or by a combination of the two. If the tower is on automatic control, the first immediate effect of changing feed preheat will depend upon the type of control system. Most present-day controls are one or the other of two general classes, viz.:

	Maintained Constant	Automatically Varied
General type No. 1.....	Reflux rate	Reboiler
General type No. 2.....	Reboiler	Reflux rate

The first immediate effect of increasing feed heat content with type No. 1 automatic system is to decrease the reboiler duty by a corresponding amount of heat. Decreasing the feed heat content increases the reboiler duty by the same amount while the reflux rate remains constant. After this first effect, the reflux can, of course, be reset for a different rate, if desired, to bring the reboiler back to its original duty or to anywhere in between.

With type No. 2 automatic system, an increase in feed heat content first increases the pumped reflux rate. Decreasing the feed heat content reduces the reflux rate while the reboiler duty remains constant. However, after this first effect, the reboiler may be manually reset, if desired, to bring the reflux rate to its original value, or anywhere in between.

The same net result on reboiler duty and reflux rate may be attained with either of the automatic systems if additional manual adjustment be made. The effect of changing heat content of feed will, therefore, be described in terms of changes in reboiler duty and reflux rate—these changes bearing the relation that, if one is changed in one direction, the other is always changed a like amount in the other direction from a heat standpoint.

It has previously been mentioned that it may be beneficial to change the feed point if there is a major change in the percent vaporization of the feed. In general, the feed point would be lowered as the percent vaporization (and temperature of feed) increases.

It may be generalized that, within limits, an increase of feed preheat at constant reboiler duty (which results in more reflux), or a decrease of feed preheat at constant reflux (which increases reboiler duty), will be beneficial to fractionation. In the first instance, the major benefit is keeping heavy ends out of the overhead, and in the second instance the major benefit is stripping light ends out of the bottoms.

If it is desired to reduce reboiler duty, say to avoid overloading the stripping trays, it may be possible to obtain the same fractionation by increasing the heat content of the feed somewhat more than the reduction in heat duty of the reboiler, and pumping more reflux.

If the reflux condenser fouls, seriously reducing the rate of pumped reflux, it may be partly compensated for by reducing the feed temperature to maintain reboiler duty at normal value, or somewhat higher. It is worth trying as a temporary expedient until the condenser can be cleaned.

If the reboiler duty falls off due to fouling or other causes, it may be possible to compensate in part for this by increasing the feed preheat to the point where the normal rate of reflux, or higher, can be pumped.

In operating a tower for the maximum feed rate, it is desirable to operate both sections of the tower at maximum loading short of flooding or serious impairment of efficiency. The same procedure will also be desirable for obtaining maximum yield of one product of given quality, or for maxi-

mum quality of both products, provided a point is not reached before maximum loading where the additional benefits do not compensate for the additional expenditure of heat. If the feed preheat can be varied, some degree of variation in the loading of each section is afforded in accordance with the load which each section will take.

Variation of feed preheat also permits determining, separately for each section, the load which causes flooding at a given feed rate. A procedure for this is outlined in the following paragraphs.

First, the load in the lower section which will cause flooding is determined by increasing reflux rate (which calls for increased reboiler duty) while feed preheat is maintained constant at the lowest possible value. When flooding occurs, the reflux is backed off just enough to bring the tower out of flooding. The reboiler duty is now as high as it can be operated for the amount of bottoms being made without causing flooding due to overloading the lower section. If the feed rate were to be increased, resulting in more bottoms, then the reboiler duty would have to be cut in order to prevent flooding.

The flooding capacity, or maximum possible load consistent with good efficiency of the upper section, will now be established. In so doing, caution must be exercised so that the maximum reboiler duty already established for the lower section is not exceeded. This calls either for a meter on the reboiler heating medium, or else a meter on the feed-preheating medium which will permit calculation of the change in reboiler duty corresponding to different amounts of feed preheat and reflux rate. The reflux rate is now cut back still further to avoid operating the reboiler above its allowable duty while the upper section of the tower is loaded up. Feed preheat is increased in small increments, the additional heat being taken out in each step by pumping more reflux. This is continued as far as auxiliaries will permit, until flooding results from overloading the upper section, or until fractionating efficiency falls off due to overload. As a last step, the reflux may be increased holding feed heat constant to bring the reboiler up to its maximum allowable duty for the lower section. If this is done, then the upper section is operating with maximum reflux for the amount of overhead product involved consistent with good operation, and the lower section is operating with the maximum reboiler duty for the amount of bottoms involved. However, if the feed rate is increased, the tower will flood at this reflux rate and reboiler duty, and it will be necessary to back off in order to avoid flooding.

It follows that a higher average feed rate can be obtained from one of constant rate than from one of fluctuating rate when a tower is being operated close to flooding condition. It also follows that any changes made while establishing flooding or in operating near flooding should be made in very small increments. In establishing flooding loads, there should be a time interval of several hours between changes, and a tower should operate 24 hours at what is considered maximum loads to make sure that small changes incidental to normal operation do not cause flooding.

As in the case of cold pump-back reflux, too cold a feed temperature cools off a tray or so at the feed point, so that the liquid will not be in equilibrium with the vapor. If the feed is charged warm enough to produce a smooth temperature gradient down the tower, the overall tray efficiency will be increased.

Optimum Pressure

As pressure is increased in a tower operating to produce a given separation, the vapor load can be increased, still maintaining the same tray efficiency. Opposing this effect is a decrease in volatility ratios of the various components with the resulting increase in required reflux quantity to effect the given separation. Consequently, for each tower making a given separation with a given feed stock, there is a pressure at which the tower should be operated to handle the maximum quantity. Conversely, at a certain fixed feed rate, there is an optimum pressure that will give the most efficient separation. Of course, due to reboiler or, principally, due to condenser limitations, the optimum pressure sometimes cannot be attained.

One company found from calculations that an 80-tray deisopentanizer tower charging debutanized natural gasoline, containing 50 percent by volume of pentanes and producing 95 percent pure *iso*pentane overhead at 95 percent *iso*pentane recovery, could be charged at the highest rate at 43 psia. Another company found, for three of its existing towers when charging an *iso*pentane-*n*pentane mixture and producing an overhead of 85 percent *iso*pentane or higher and a bottoms of 5 percent *iso*pentane, that 30 psia or lower, was optimum. The optimum pressure did not appear to be the same for each of the towers which had different numbers of trays. The tower with the lowest number of trays appeared to have a lower optimum pressure than the other towers. A similar investigation by a third company on a deisobutanizer tower of 50 plates charging crude alkylate, and producing 81.7 mole percent *iso*butane in the overhead and 1.9 mole percent i-C_4 in the bottoms, showed that the optimum pressure for maximum charge rate was 108 psia.

In the cases that have been investigated where the data are available, it appears that the optimum pressure occurs in the range where the condenser may limit the capacity rather than the tower size. If this be the case for other types of separations, it would be economical to run the tower at different pressures according to the seasonal variations in cooling-water temperature. In the colder months, the optimum pressure could either be attained or approached more closely than in the warmer months. With this scheme of operation, an improved fractionation or increased capacity could be attained during part of the year rather than being limited the year-round by the pressure that has to be used during the hot months.

Instead of seasonably changing the operating pressure in a tower, in some cases it might be preferable to operate the tower at the same pressure throughout the year. This pressure, however, would be as close to the optimum as possible as determined by the overall yearly yields and purities of the desired products and by equipment limitations. Under this scheme, the sharpness of separation would be seasonably varied to permit tower operation near the optimum pressure, in the summer the fractionation being poorer than in the winter.

Towers in Parallel

In the case where two towers are operating in parallel, each taking a part of the same feed and producing a combined product of given specification and yield, it may happen that one tower has more trays than the other, and each tower may have different operating characteristics in regard to the increase of reflux per barrel of feed to increase of product purity. The tower with fewer trays may be operating so that a much larger change in reflux per barrel of feed is required for a given change in product purity than is the tower with more trays. A higher combined feed rate may be possible when operating the former to produce a product of less than specification purity, and the latter to produce a product of higher than specification purity, than can be obtained by operating both towers to make the same specification product. The tower which is making under-specification product is charged with a higher proportion of the total feed than when both towers are making specification products. Also, this same procedure can be used if the two towers have the same number of trays but different tower diameters. The smaller tower would be charged at a rate to produce a sloppier cut than the larger tower produces.

Absorption Systems

In absorbers operating for recovery of butane and heavier, the butane absorption increases with oil circulation up to a point beyond which very little additional butane is absorbed, but a considerable amount of propane and lighter continues to be absorbed. This must be stripped out of the rich oil and eliminated from the system by one means or another. The net result, in some cases, of circulating more than the minimum critical amount of oil is to reduce the overall butane recovery due to losses associated with elimination of the additional propane and lighter which was absorbed, or reduction of capacity.

The gas capacity of absorbers may be increased, or the oil circulation reduced for given capacity, by using an absorbing oil which has the lowest viscosity and the lowest molecular weight for the vapor-pressure requirements.

Typical Control Systems

In the operation of fractionating columns, the problem of control is one that must be given considerable study, as a fractionating column can operate with efficiency only with good control. Figure 9 through 12 show a number of standard methods of control. It is not the purpose of this paper to give all the possible methods of control, but only some of those in common use. Also, it is not intended that these control setups be taken necessarily as the proper combination for any one setup. For instance, the type of control system shown in Figure 10 for kettle control might be combined with the system shown in Figure 11 for pressure control,

and other combinations might be worked out to meet the specific problems of any given setup.

Figure 9 shows a typical instrumentation used in a fractionating column producing constant-vapor-pressure bottoms. The vapor pressure of the bottoms is defined by the temperature and pressure in the kettle. Therefore, it is desirable that the pressure tap for control be taken from the kettle. As the

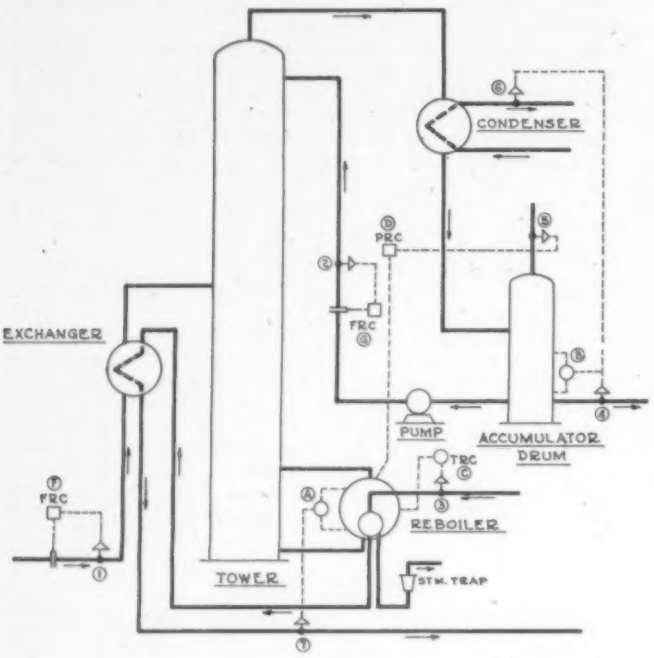


FIGURE 9
Tower Control Systems

composition of the bottoms is defined, the composition of the overhead must vary to accommodate variations in feed composition. To allow this condition to take place, no attempt is made to control the column-top temperature.

The reflux is controlled by a flow-rate controller. The bottoms product is controlled by a liquid-level controller with a motor valve in the outgoing fluid line. If the bottoms product from the column is used to heat the feed by means of exchangers, it is necessary that the streams be maintained as nearly constant in rate as possible in order not to vary the heat input to the feed. Referring to Figure 9, which shows such an arrangement, it is usually advisable to use a valve positioner in valve No. 7 with a sensitive-type liquid-level controller **A**. Should the column be operating without a heat exchanger and feeding directly into another column, it is advisable that the same precautions be taken, on the liquid-level controller and motor valve as outlined previously.

It is generally considered good practice to put the motor valve controlling the flow of bottoms from the column on the downstream side of the heat exchangers and the motor valve on flow-rate controllers on the downstream side of the orifice.

Motor valve No. 3 controlling the heating medium to the reboiler operated by the temperature controller **C** should be made as nearly frictionless as possible through the use of a valve positioner. In a number of cases the preference is to use flow-rate controllers operating the motor valves, the flow-rate controllers being reset by the liquid-level controller or the temperature controller. Even if flow-rate controllers are used, better operation is obtained by the use of valve positioners as well.

It will be noted that in all these illustrations the feed to the column is shown as being controlled by a flow-rate controller **F** actuating motor valve No. 1. Such a setup is usually desirable but often, due to other plant conditions, this type of feed-rate control is not applicable. However, in order to operate a column at high capacity and with high efficiency, it is necessary that the feed rate be maintained essentially constant.

In Figure 9, if substantially all of the net overhead is condensed and withdrawn through valve No. 4 (operated by liquid-level controller **B**), and a very small amount of fixed gas is released through valve No. 5, poor pressure control will result. If this condition exists, the type of pressure control shown in Figure 12, either with or without a pressure controller **E** and motor valve No. 8, would be applicable.

In Figure 9, if it is not desired to produce liquid overhead product, liquid-level controller **B** can be connected to motor valve No. 6 in the water line to the reflux condenser, so that only sufficient condensation takes place to produce the desired reflux, the overhead product passing off as vapor through valve No. 5.

The control problems for columns making a substantially pure overhead product are somewhat different. Under this condition the overhead-product composition is fixed, and the bottoms product must vary as the feed composition varies. Therefore, a definite bottoms temperature with a constant pressure cannot be maintained. Under this condition, a constant column pressure can be maintained and the controls arranged so that the bottom temperature may vary to accommodate variations of feed. Figure 10 shows one method accomplishing this result. Temperature controller **C** has its bulb located in the column between the feed and the bottom at the point well within a good break in the temperature gradient. Reference to Figure 5 will show this point more clearly. As this is a point of definite composition in the column, the pressure control should be taken from this point or very close to this point. The type of pressure control shown in Figure 10 is satisfactory and very sensitive where the rate of liquid withdrawal through valve No. 4 is greater than approximately one-eighth of the reflux pumped back over the top of the column. If the liquid withdrawal through valve No. 4 is very small with respect to the amount of reflux, this type of control becomes very insensitive, and relatively very large variations of pressure may result. Under these conditions the type of control for pressure shown in Figure 12 is preferable.

Figure 11 shows a kettle controlled by the temperature taken within the break point in the temperature gradient between the top of the column and the feed. This type of control is very satisfactory provided there are not too many trays between the point of temperature control and the kettle; otherwise, the lag between the control point and the point of heat application is large, requiring a large throttling range on the temperature controller to prevent hunting. This causes the control to lack sensitivity, and it will wander far from the control point due to changes in feed rate and feed composition.

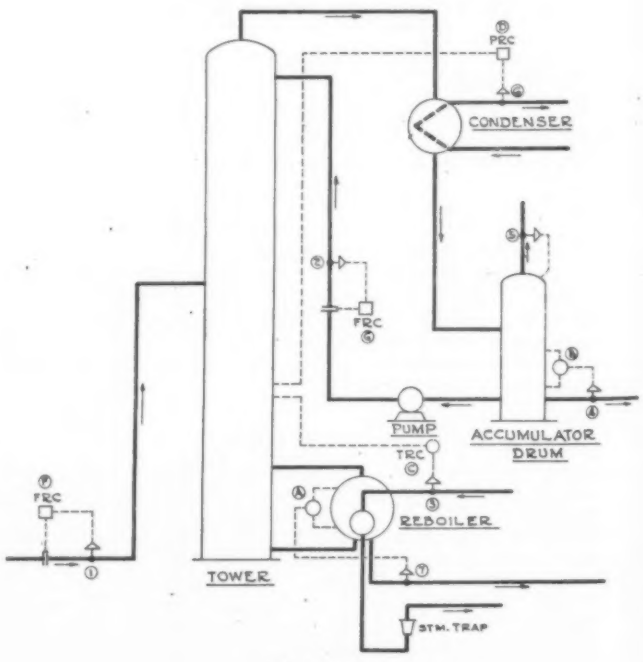


FIGURE 10
Fractionating Column Producing Pure Product Overhead. Overhead Totally Condensed. Case 1.

The type of pressure control shown in Figure 11 depends on flooding the reflux condenser so that only sufficient surface is exposed to vapor condensation to give the desired rate of condensation. This method of control is subject to the same comments as given for the pressure-control system shown in Figure 10. This control system is also subject to the further criticism that, if there is considerable excess sur-

face in the reflux condenser and water temperatures are low, the reflux going back to the column will be substantially subcooled. This will result in converting some of the top trays in the column from fractionating trays to heat-exchange trays, with the overall result of reducing the total number of effective trays in the column.

Figure 12 shows a control system where no attempt is made to control the kettle temperature. This column is shown operating with a constant heat input to the kettle, and the reflux quantity is controlled by the temperature at the break point of the temperature gradient in the top part of the column. This type of control maintains a substantially constant vapor rate through the column, irrespective of normal changes of feed rate and feed composition. The type of pressure control shown in Figure 12 is applicable to shell-and-tube type reflux condensers, and also to atmospheric sections in a cooling tower or submerged sections in a box-type condenser.

The main column pressure control operates valve No. 9 in the vapor line between the column and the reflux condenser. Pressure controller **E**, operating valve No. 8, is used to maintain a pressure a few pounds below the column operating pressure in the reflux accumulator. By bypassing hot vapors from the top of the column into this accumulator, the temperatures of this accumulator will be raised above the temperature of the liquid coming from the reflux condenser. By maintaining the pressure in the reflux accumulator constant, a constant differential pressure is maintained across the reflux pump, thereby giving far more stable operation to this piece of equipment.

General Principles in Selecting Proper Control System

There are certain general principles to be kept in mind in selecting the proper method of control of any process. This point of control should be selected at a point where the quantity of material handled is large, or where the change of temperature is large with respect to the change of temperatures at other points. An example of control where quantities are large will be the use of a motor valve in the vapor line between the column and the reflux condenser, as shown in Figure 12. A case where the change in magnitude

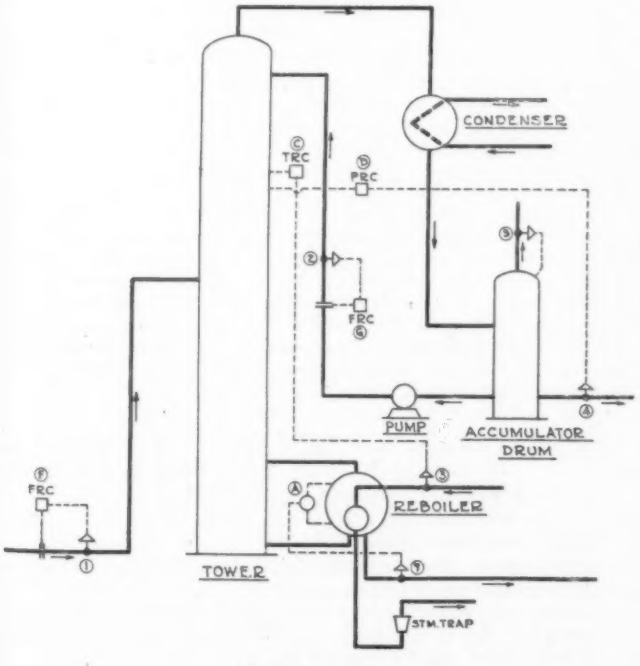


FIGURE 11
Fractionating Column Producing Pure Product Overhead. Overhead Totally Condensed. Case II.

is large at one point with respect to the change in magnitude at another point is taken advantage of in the control shown in Figure 12 for the temperature in the top part of the column. If the point of control is properly selected, temperature change at this point of 5 degrees will only represent possibly half-a-degree change at the column top. Therefore, the sensitivity of control is greatly improved. Instruments should be so selected and located that their speed of response is at least equal to the rate of change of feed compositions or feed rates. The water temperature to reflux condensers, steam pressure, or heating-medium temperature is always subject to some variation. In order to get proper control of the column, the instruments must be so adjusted that they will keep in step with these rates of changes of the utilities.

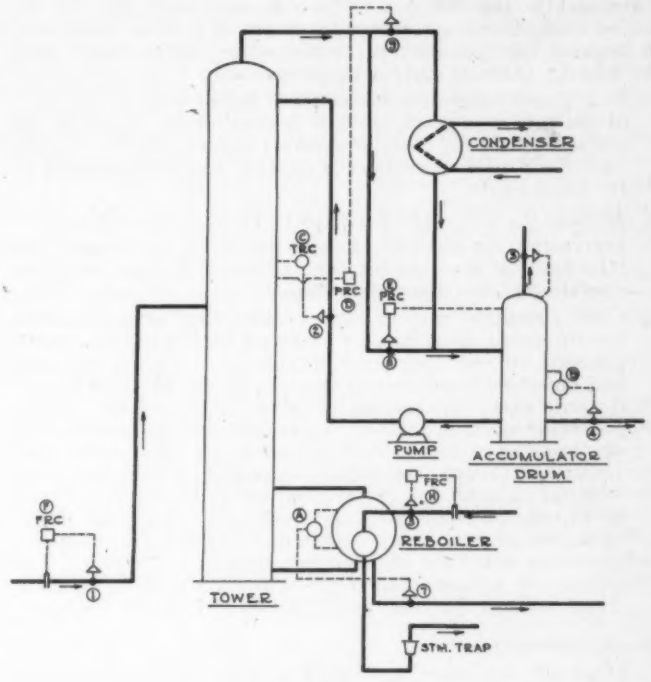


FIGURE 12
Fractionating Column Producing Pure Product Overhead. Overhead Totally Condensed. Case III.

If the response of the instruments is too rapid, cycling conditions will result. These cycling conditions are due to lags.

One type of lag is that due to inertia of a large body of liquid passing through the system. If there is a long line full of liquid traveling at a relatively high velocity, and if an attempt is made to throttle this liquid, the inertia of this liquid will tend to maintain a constant velocity even with considerable throttling. Finally, the velocity is checked and the motor valve is closed beyond the point at which proper flow normally could be obtained, resulting in overshooting the mark.

The other type of lag which is encountered is temperature lag. In a column there is a large amount of metal as well as liquid that has a relatively high heat capacity. When any change of temperature occurs, the liquid and the metal must be readjusted to the new temperature condition. If the response of the instrument is too fast, over-control will result, and the system will start and continue to surge. In a great many processes, due to variations of cooling-water temperature and other causes, feed composition will vary from time to time. If instrument response is too slow, these feed-composition changes can produce surging and unstable operation throughout the process.

Steam Traps

There are a number of types of setups to remove steam condensate from the tube bundles and kettles or other heating equipment. The methods in most common use are steam traps, continuous drains, or pots with liquid-level controllers. All methods give satisfactory operation if certain precautions are taken. The inverted bucket-type trap will remove from the system all fixed gas that may enter with the steam. A steam trap, if not properly installed, will produce a temperature fluctuation each time the trap dumps. This is usually caused by the trap not being set sufficiently below the tube bundle, and by the condensate line from the tube bundle to the trap being too small. The use of a pot with a liquid-level controller or a continuous drain will give smooth operation, but may cause trouble due to gas binding in the tube bundle. This can be overcome by installing a small line from the top of the condensate pot to the condensate line bypassing the

motor valve. If a small amount of steam is continuously withdrawn through this bypass, fixed gas will be removed continuously from the system.

CHANGES OUTSIDE TOWERS

Reboiler Trouble

1. *Small Reboiler Surface*

Frequently the reboiler surface is too small for the required load. There are several methods that have been used to increase the load on the reboiler when the temperature of the heating medium cannot be increased:

a. If a thermo-syphon reboiler takes liquid from the bottom of the tower, in some cases it is possible to rearrange the piping so that the reboiler receives liquid from the bottom tray. This scheme reduces somewhat the temperatures of the heated side.

b. Reduce the pressure in the tower as far as possible.

c. Increasing the amount of vaporization of the charge will take load off the reboiler. The effect of this on the tower operation is discussed elsewhere.

d. One company injects water into the heated-medium stream going to a reboiler in order to reduce the partial pressure of the hydrocarbon, thereby reducing the inlet and outlet temperatures of the reboiler by 50 to 70 degrees in some cases. In the case of debutanizers and also depropanizers, when producing 400-end-point bottoms product, the injected water will all condense on the bottom tray. Inasmuch as any corrosive compounds contained in the bottoms product will dissolve in the water, the water must be withdrawn continually and fresh water reinjected. A part of the water may be recirculated. Provision for combating corrosion will have to be resorted to in some cases. This process is protected by U. S. Patent 2,325,839, issued to Eastman and Scoville, assignors to The Texas Company.

2. *Vapor Binding*

There are two ways in which a reboiler can become vapor-bound with a resulting marked decrease in reboiler load. The most serious type of vapor binding is that on the surface of the tubes exposed to the heated medium. This is brought about by using a heating medium too high in temperature, causing such a rapid heating and vaporization of the heated material that a continuous film of vapor is formed on the tubes which effectively decreases the overall transfer rate. This phenomenon occurs when the heat density is higher than about 20,000 to 24,000 Btu per hour per square foot. If the heat density is well above this figure, the resulting transfer rate will be low and the reboiler will operate without surging. However, if the reboiler is near the vapor-binding condition, it may surge, as the transfer rate will vary depending on the amount of vapor binding in different sections of the reboiler. The surging will be evident by fluctuating reboiler outlet temperature. The temperature will continually dip below the required outlet temperature. This condition can be eliminated by decreasing the mean temperature difference by lowering the heating-medium temperature to the point where the surging stops.

Vapor binding in the heating medium can be caused by the presence of air or non-condensable gases which reduce the transfer rate appreciably. With hot-oil heating medium, vent valves should be provided so that the gases may be vented. With steam, the trap or condensate-control valve may be bypassed to purge the steam chamber, or a continual small bleed stream of vapors from the chamber can be provided. Non-condensable gases frequently are found in steam systems resulting from the water-treating methods used. Also, air sometimes is drawn into the reboiler steam chamber through the condensate trap if the condensing pressure is close to atmospheric pressure. The trap will not operate properly under these conditions. If this is the case, the condensate piping can be so arranged that the bottom tubes are kept flooded with water, thereby blanketing these tubes. The consequent effective reduction in reboiler surface will raise the condensing temperature and pressure of the steam automatically. In order to transfer the same amount of heat, a greater temperature difference will be required to make up for the effective decrease in reboiler surface. Another type of vapor binding in hot-oil heating systems is brought about by flashing some of the hot oil, on passing through the control valve, upstream to the reboiler. These vapors effectively lower the transfer rate which, in most cases, will cause fluctuations of reboiler operation. Increasing the oil pressure at the outlet of the reboiler or moving the control valve to the outlet side of the reboiler will eliminate the difficulty.

3. *Surging*

The reboiler construction may cause surging. Too close tube spacing in kettle-type reboilers or horizontal thermo-syphon reboilers will not allow smooth vapor flow. Improper baffle construction also will cause vapor surging in these types of reboilers. In most of these cases some tubes or baffles may be removed without harming the overall transfer rates. Also, in some horizontal reboilers, sufficient vapor space is not present above the tubes, and surging will occur. Increasing this space or providing larger outlet nozzles will eliminate difficulty.

4. *Pressure Drop*

Another possible source of trouble with thermo-syphon reboilers is too small inlet and outlet lines or too high pressure drop through the reboiler, causing liquid to back up to the bottom tray. This trouble may be eliminated by increasing the line sizes or by removing the bottom tray to allow more available head to the reboiler. Also, in horizontal reboilers with tubes too close to the inlet a restriction to flow is present. Removing several tubes will eliminate this difficulty.

Line Sizes and Vapor Binding

1. *Vapor Overhead Lines*

Excessive pressure drop in the overhead vapor line between the top of tower and the reflux condenser has two harmful effects:

a. It causes the pressure difference between the pressure-control point on the tower and the condenser to vary appreciably with changes in the amount of vapor flowing through the overhead vapor line. Each time this occurs, the automatic pressure controller must readjust itself, which tends to make pressure control uneven—giving undesirable surges within the tower, which also upsets temperature control.

b. It necessitates operating with a lower condenser pressure which, in turn, requires more condenser surface or more or colder cooling water. Tower systems have been known to be bottlenecked solely by excessive pressure drop in the overhead vapor line, which introduced an unnecessary temperature spread between the reboiler and the condenser. The excessive pressure drop in the overhead line may be due to small line size, or to a pressure-control valve in the line taking too much pressure drop. The remedy is obvious.

2. *Reboiler Lines*

If the tower nozzle and the lines feeding liquid from tower to reboiler are too small, or if the vapor return line between reboiler and tower is too small, the result may be tower flooding, or a limitation of reboiler duty due to an increase in reboiler outlet temperature caused by the bypassing, around the reboiler, of some liquid from the bottom tray.

Fouling on the cold side of the reboiler will produce the same result in certain types of reboilers; hence, it must be established whether such troubles are caused by insufficient line capacity or by fouling of the reboiler. In many cases, fouling can be detected by a poor heat-transfer rate on the reboiler. When flooding occurs due to reboiler trouble, the tower starts filling with liquid from the bottom up, and it may be possible to detect this condition by a comparison of pressure drop across various trays, as obtained from gauge glasses or other means.

3. *Water Lines*

Condensing capacity and reflux may be bottlenecked by inability to get enough through the condenser due to the water lines being too small for the amount of available pressure on the water.

4. *Control-Valve Sizes*

Control valves which are too small may introduce excessive and unnecessary pressure drop through lines which, in some cases, imposes limitations on tower performance. However, if the control valve is too large, poor control will result. Temperature-control valves should be sized most carefully. In general, the control-valve pressure drop should be a minimum of 20 percent of the total pressure drop in the system.

Condenser Arrangement

In cases where there is series flow through the hot side, the cold side, or both sides of condenser sections, excessive pressure drop may occur—causing a limitation in condenser capacity. This might result due to necessity for operating at a lower pressure, or to inability to get sufficient water through the condenser. The capacity of the condenser may be increased by rearranging the sections for parallel flow on the side where pressure drop is excessive.

In condensing a liquid where little change in temperature

is involved, as in the case of pure components, or a mixture of very close-boiling components, internal baffling on the hot side of the condenser is not necessary. If there are closely spaced internal baffles, the pressure drop through the hot side of the condenser may be reduced by respacing the baffles as far apart as is consistent with the tube supports.

Operating Changes

1. Bypassing Vapors Around Tower

At times it is possible to increase the apparent capacity of a fractionating column by flashing the feed and bypassing the vaporized portion around the column, thereby relieving the load on the fractionating equipment. This has been successfully accomplished in re-running alkylate. It has been found that, if approximately 50 percent of the alkylate is flashed off prior to entering the fractionating column, the alkylate produced will have the same characteristics as if all the alkylate had passed through the fractionating column. As this alkylate has a very narrow boiling range, it is practically impossible to control the percentage of vaporization by temperature.

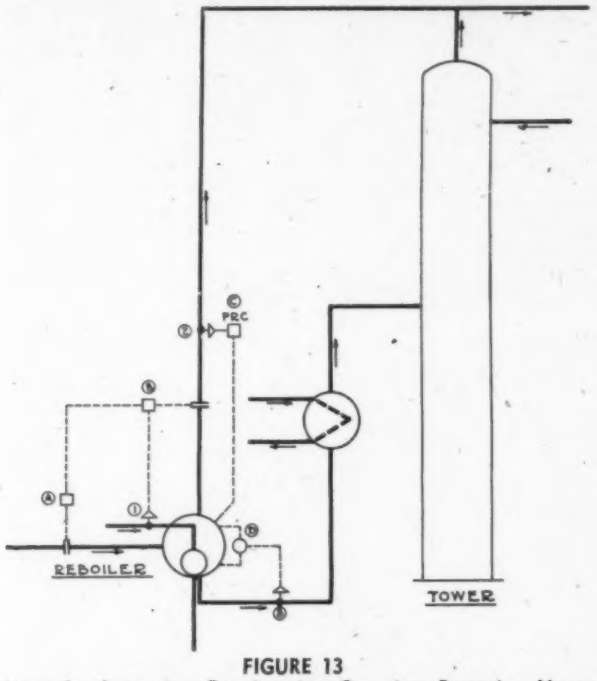


FIGURE 13
System for Increasing Fractionation Capacity—Bypassing Vapors Around Tower.

Figure 13, shows the method of accomplishing this controlled vaporization. The unvaporized feed enters the flash chamber through flow rate controller **A.** The vapors leave the flash chamber, pass through the flow controller **B** which is reset by flow controller **A.** Flow controller **B** operates motor valve No. 1 in the heating medium to the flash chamber. Pressure controller **C** maintains a constant pressure on the flash chamber by controlling motor valve No. 2 in the vapor line between the flash chamber and the vapor line from the top of the column. Liquid-level controller **D,** operating motor valve No. 3, withdraws the unvaporized portion of the feed through the preheater into the column.

There are a number of cases where, due to the heavy nature of the base stock, substantial vaporization can be taken on column feeds without materially affecting the purity of the column overhead product. This method of vaporization also can be used to advantage on a narrow-boiling-range feed stock to a column where it is desired to maintain a constant percentage of feed vaporization.

2. Rearranging Order of Fractionation

The overall capacity of a system of fractionating towers often can be increased by changing the order of fractionation, taking advantage of variations in size of this equipment to produce the desired cuts.

3. Taking Side Cuts

In a number of instances where the overhead product from a column passes into other columns for further fractionation, it often is possible to reduce materially the quantity of bottom products from the second column by taking a side stream from the preceding column. This side stream can be withdrawn at a point in the column and at such a rate that, when it is combined with the bottoms product from the second, the amount of light overhead product in the resultant stream does not materially exceed the normal content of this product in the bottoms from the second column when not withdrawing a side stream. Also, this side-stream product must not contain a sufficient quantity of the next heavier component to damage the bottoms product from the second column. By taking a side stream from the first column, the kettle temperature in the succeeding column will be reduced—thereby greatly increasing the effectiveness of the reboiler, both from a heat-input standpoint and from a stripping-duty standpoint. The stripping duty is principally reduced by reducing the quantity of bottoms product from the second column.

The principle may be applied in much the same way when the bottoms from a column receives further fractionation in a second column, in which case the side stream from the first column would be combined with the overhead from the second column. Additional reboiler capacity thus is made available in the second column. The amount of additional capacity is that formerly required to vaporize the fraction withdrawn as a side stream from the first column.

A side stream often is useful in an existing fractionation system when it becomes necessary to make sharp cuts on both ends of a column operating at an excessive rate. The side stream is used in this instance as a slop cut.

4. Feed Conditions

As pointed out previously herein, the conditions of the feed, i. e., its proper temperature, percent vaporization, and feed introduction into a column, can have considerable effect on the overall tower capacity and efficiency. Therefore, this problem should be given serious study before deciding that equipment is too small or has reached its limit of fractionating efficiency.

In a number of cases, one column is fed with two materials of substantially different composition. By introducing these two streams separately into the column, and under proper conditions of temperature and vaporization for the points of feed introduction, additional capacity and fractionating efficiency can be obtained. In some cases, where a very small percentage of the feed is taken off as bottom product, an unstable operating condition results. The column has a tendency to surge, thereby reducing capacity and fractionating efficiency. This condition often can be corrected by recycling a portion of the bottoms product so that sufficient liquid is withdrawn from the bottoms at all times to maintain stability. In order properly to strip the bottoms under these conditions, the reflux rate and heat input to the column may have to be increased.

One cause of poor stability due to too small a percentage of bottoms product is insufficient liquid on the trays below the feed. Trays, in general, have a minimum liquid rate at which they will operate efficiently. There are cases where the normal liquid rate through a column is less than the weep-hole capacity of the trays. As a result, the trays are running dry, and no real contact between the descending liquid and rising vapors is obtained.

Changes Inside Towers

Level Trays

In order to maintain efficiency in a column, it is necessary to have an equal quantity of vapor rising from each cap and an equal quantity of liquid passing each cap. One of the chief requisites for even distribution over the tray is levelness. Great care should be exercised to see that all the trays are level. All the caps in a row likewise must have the same submergence. If the tower has a high hydraulic gradient across the trays, the caps nearest the inlet downflow must be set higher than the caps next to the outlet downflow. If the overflow weirs on the tray are not level, improper distribution of liquid across the tray will result, as most of the liquid will flow to the low point of the weir. Therefore, the overflow weirs on a tray should be straight and level. It sometimes is necessary to install some method of distributing the liquid, especially at high liquid rates, on the inlet side of the tray.

Inlet Nozzles

In a great many columns it is the practice to introduce the feed at almost any point on the circumference of the tray. If the column has an excess number of trays, this practice may not show any material decrease of fractionating efficiency. However, if the column is operating close to the minimum number of trays, it is important that the feed be introduced in such a way that the liquid flow across the trays is not upset.

One method of accomplishing the feed introduction without upsetting the flow across the tray is shown in Figure 14. The liquid is introduced through the feed nozzle against a baffle which deflects the liquid portion of the feed into the down

spout from the tray above the feed. Vapors in the feed are released into the space between the tray above the feed and the tray second above the feed. In this manner, the liquid flow is not disturbed on any of the trays. The objection to this method of feed introduction is that the tray above the feed is bypassed by the vaporized portion of the feed, but it is believed that the loss of vapors through this tray is not so

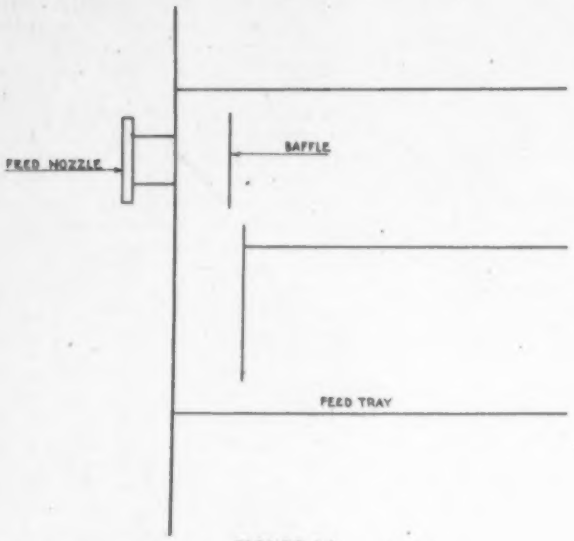


FIGURE 14
Method of Feed Introduction

damaging to the tower operation as the disturbance of liquid flow with its resultant channeling. However, if the charge is introduced at a temperature considerably higher or lower than the charge-tray temperature, putting the charge into the downflow may cause vaporization in the downflow, resulting in vapor binding and flooding. In this case, the charge should be introduced in the center of the tray.

Cap and Uptake Layout

When the number of caps is in excess of that required to maintain good slot velocities, and when the gas pressure drop is low compared to the hydraulic gradient, some caps and risers should be removed. A guide for removal follows:

1. If the space between the downflow from the tray above and the nearest cap is less than 3 inches, and no vapor dam or blanked slots are present, the row next to the downflow should be removed. If the caps are closer, there is a possibility that they will blow gas up inside the downflow, breaking the seal and causing the liquid to pour down the risers. Once this process starts, the tower operation will be stable, but the gas going up the downflows and liquid down the risers will result in poor fractionation.
2. The row next to the weir should be 3 inches away from the overflow weir. If the caps are closer, they will blow liquid off the tray, and the actual static submergence will be considerably less than desired. The larger this space, the better will be the vapor disengaging of the liquid entering the downflow.
3. Caps which are less than 1½ inches apart are undesirable. Caps closer than this will cause the vapor to channel more easily through the liquid.

Removing caps will decrease the liquid gradient; but care must be taken that channels are not created, resulting in poor liquid distribution.

If it is discovered, by calculation of the hydraulic gradient of clear liquid, that poor vapor distribution is present even though the slot velocities are high, the following changes can be made:

1. Raise the caps higher from the tray at the inlet end of the tray to compensate for the liquid gradient. Other rows of caps may be raised but to a smaller degree.
2. Provide restrictions in the uptakes next to the outlet end to compensate for the small liquid head that the gas has to bubble through at this end. Providing restrictions makes a more stable tray, and will make it possible to have a static seal of all the slots and the weir which might not be the case if some of the caps are raised.

The foregoing comments apply principally to cross-flow trays with round, hexagonal, or similar types of caps. In the case of trays with circular downflows distributed over the surface of the tray, the liquid gradient is usually small, because the length of travel is short. Trays with long, rectangular caps also have small liquid gradients. However, with these trays, caps should not be placed too close to downflows.

In cases where the possibility exists of blowing gas up the downflows, a weir can be installed upstream to the first row. Care must be taken to leave sufficient space for the liquid to flow over the weir from beneath the downflow. Another way of accomplishing this is to blank off the slots next to the down-flow. The slots on the caps next to the overflow weir can also be blanked off to prevent blowing the liquid off the tray.

Holddown bars located on top of caps appear to offer a large resistance to flow, and will cause large liquid gradient even if the clear liquid level is below the bars. In the case of oblong caps where the flow of liquid is straight across the plate, the holddown bars should not affect the liquid gradient nearly so much if they are located parallel to the liquid flow. Holddown bars should be located, if possible, underneath the tray so that they cannot offer resistance to liquid flow in any manner.

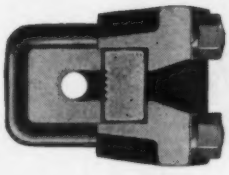
In some cases the length of an overflow weir is so short that the height of liquid flowing over is large and the gas-pressure drop through the trays is high. It might be undesirable to lower the weir to reduce the liquid head on the tray, as a negative static seal might result. In this case it might be possible to remove the row of caps next to the weir and replace the straight weir by a zigzag weir, thereby increasing the effective length of the weir upwards of 30 percent.

In some trays the constriction area in the downflow to the tray below should be increased by removing a portion of the bottom of the downflow.

When the caps are cast-iron, they can easily be replaced with gauge metal caps, with a resulting increase in tower capacity. The capacity increase is accomplished by appreciably lowering the hydraulic gradient and by decreasing the pressure drop between the risers and caps. A much smaller increase in tower capacity is realized when the risers are also changed from cast-iron to gauge metal.

The Appendix—Study of Alkylation-plant Isobutane Tower Performance—will appear in an early issue.

Alike in Quality



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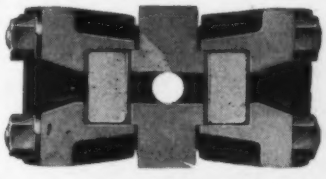

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OPERATION HOW TO DO IT MAINTENANCE

Steam-Turbine Drive Cuts Tube-Rolling Cost

ARKANSAS Fuel Oil Company's gas-treating plant at Columbia, Arkansas, sweetens and strips gas containing a high percentage of hydrogen sulfide and carbon dioxide. This gas causes severe corrosion in heat exchangers, reboilers and reactivator of the purification unit, and replacement of tubes in reboilers and heat exchangers is frequent. The tube bundles used contain 800 to 900 ¾-inch carbon-steel tubes, and to drill out and roll in this number of tubes is a tedious task. The plant did not have sufficient air capacity to operate an air-driven roller; also it was found an air-driven motor often caused the tube rollers to break.

G. E. Freeman, chief maintenance mechanic at this plant, devised a means of speeding tube replacements. He built a steam turbine by cutting a wheel out of ¼-inch steel plate and welding vanes on one side of it. The housing was made by cutting a ring off of a joint of 16-inch pipe and welding a flat plate on one side. A flange welded to the other side of the ring and a flat steel plate bolted to this flange incloses the wheel in the housing. Jets welded on a piece of ½-inch pipe are inserted in the housing is such a way as to propel the turbine wheel with steam. Two automobile generator bearings on each side support the turbine shaft. This turbine is mounted on a frame and connected to an automobile transmission with a belt drive. A 6-foot ¾-inch flexible drive shaft is attached to the other end of the transmission box, with a tube roller fitted to the end of the shaft.

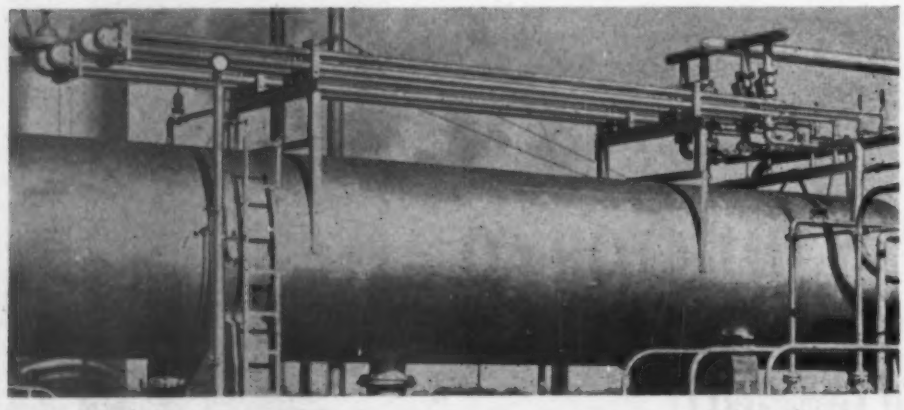
By operating the transmission in high gear, the tube roller works satisfactorily rolling in. When tubes are rolled sufficiently, the transmission is reversed and the roller backed out of the tube. The reversing operation is accomplished by pulling a cord attached to the shift lever. An "S" shaft slot fitted over the shift lever causes the lever to move from high gear to reverse, and vice versa. A spring attached to the top of the shift lever holds it in high gear when the cord is released. The operator needs to pull the cord only when the tube is rolled sufficiently.

Steam turbine, automobile transmission and flexible drive set up for tube rolling.

A friction clutch prevents rolling the tube too much. This clutch is a pulley fastened to a sleeve with a disc on one side. The pulley and sleeve turn free on the turbine shaft but are held against another disc by a spring. When the tube is rolled enough, these discs start slipping. Tension on the spring determines the load the drive shaft will pull.

A morse tapered sleeve attached to the end of the flexible shaft and a socket welded to a morse tapered shank to hold the tube roller makes it convenient to remove the roller from the shaft or to disengage the roller while the machine is in operation.

This device has cut the labor cost of rolling tubes more than half. One man using it can roll tubes as fast as two men can put them in place in the bundle.



These fin type condensers for depropanizer overhead were mounted on top of a raw-make tank in a gasoline plant to eliminate foundation costs, conserve space, provide gravity flow, and simplify piping. Each condenser is equipped with thermometers and control valves for the water and vapor.

Liquid-Level-Gauge Piping on Process Tanks

A TANK reworked for use as a lean-oil surge tank required a liquid-level gauge, which was constructed at the time the tank was reworked from the materials at hand. As shown in the photograph, 2-inch and 1.5-inch pipe was used and the gauge glasses were given a 90° spread. As the tank is open to atmosphere, it was not necessary to use a shutoff valve on the top leg. The tank is equipped with welded nipples at top and bottom points into which the 1.5-inch pipe nipple is screwed. Connections are made to the 2-inch pipe leg to which the gauge-glass connections are made.

The same method was used in equipping a pressure vessel (raw-make tank) with gauge glasses, with the exception that valves were used on both top and bottom connections, so that the leg can be removed at the unions for cleaning or replacing the glasses. In addition, a connection is supplied at the top of the leg for an indicating pressure gauge, and

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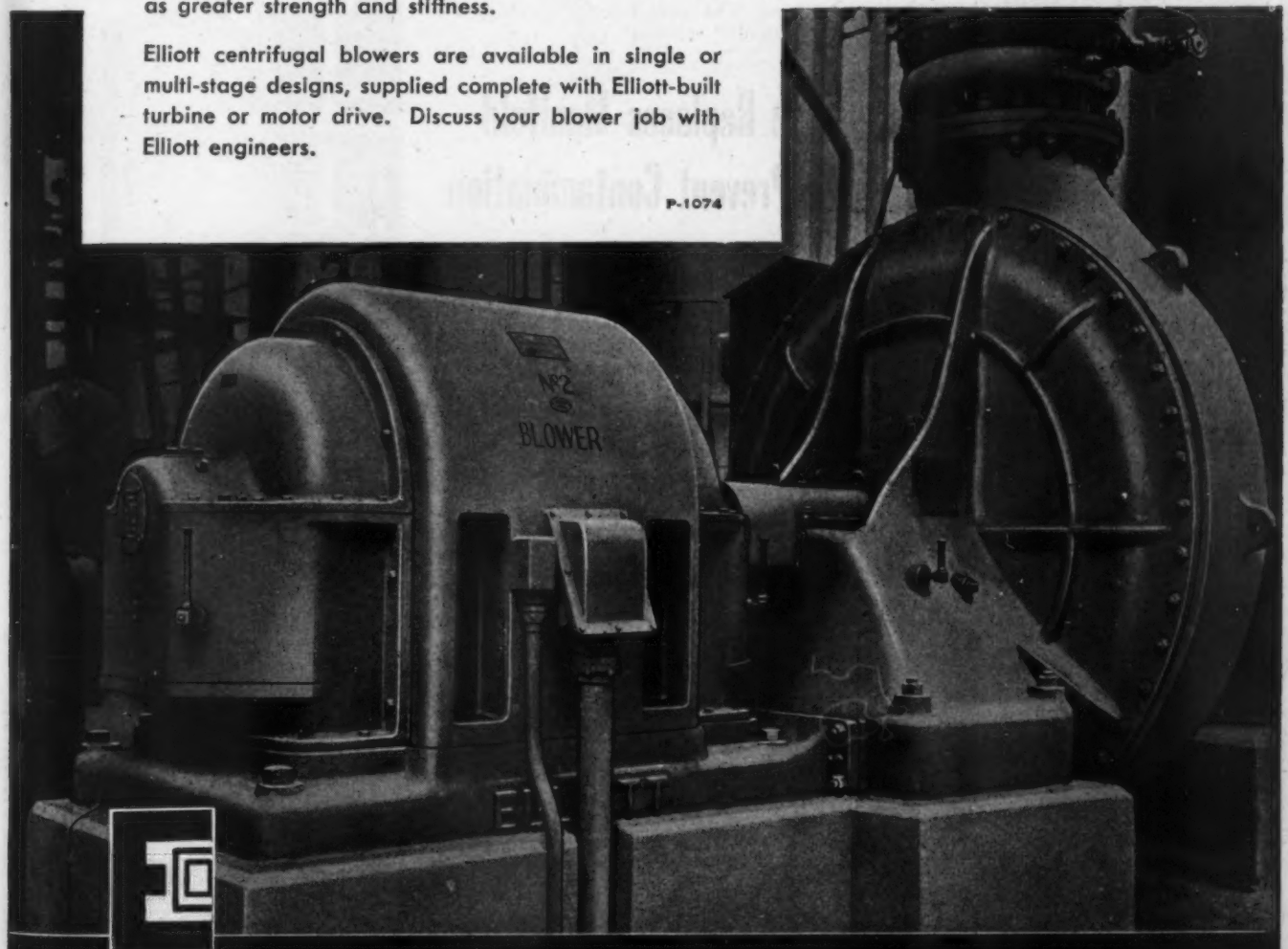
P-1074

Below is shown an outdoor installation of an Elliott motor-driven blower, typical of many used in catalytic cracking service. Blower supplies 21,500 cfm of air at 2.5 psig. Driven by 350-hp, 3550-rpm motor.



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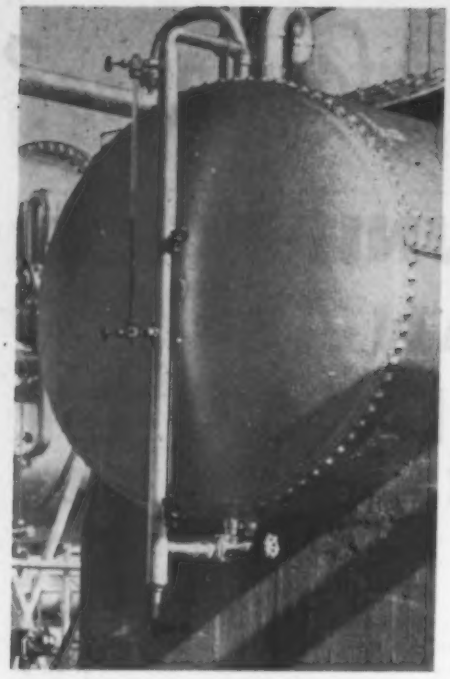


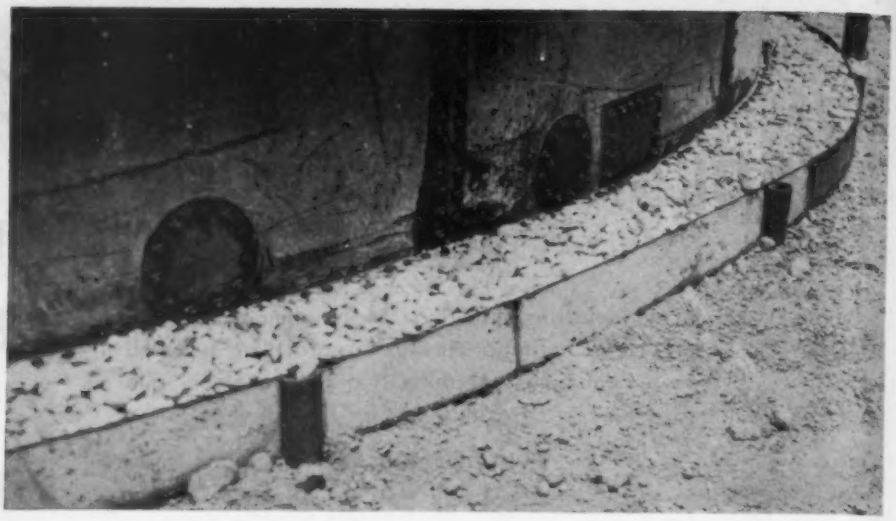
Gauge glass installation.

at the bottom for a small valve to drain the water from the gauge leg to eliminate the U-tube effect for a corrected reading.

Vessels for Small Plant Fabricated From Pipe

DUE to the availability and delivery schedules for prefabricated equipment, this fat-oil flash tank, used in connection with the reabsorber in a small gasoline plant, was constructed from a 16-foot length of 23-inch pipe. Dished heads were welded to each end. By using the tank in a horizontal position, the large surface provided makes operation satisfactory.

The 12-foot accumulator shown at the right was fabricated from 23-inch pipe, also. Bull plug welded ends were used instead of dished heads. The bootleg beneath the tank was made from a length of 8-inch pipe with an orange-peel welded bottom and a flanged head for connecting to the tank. A gauge glass and drain cock were added to drain the water from the tank. The vessel is satisfactory for low-pressure service.

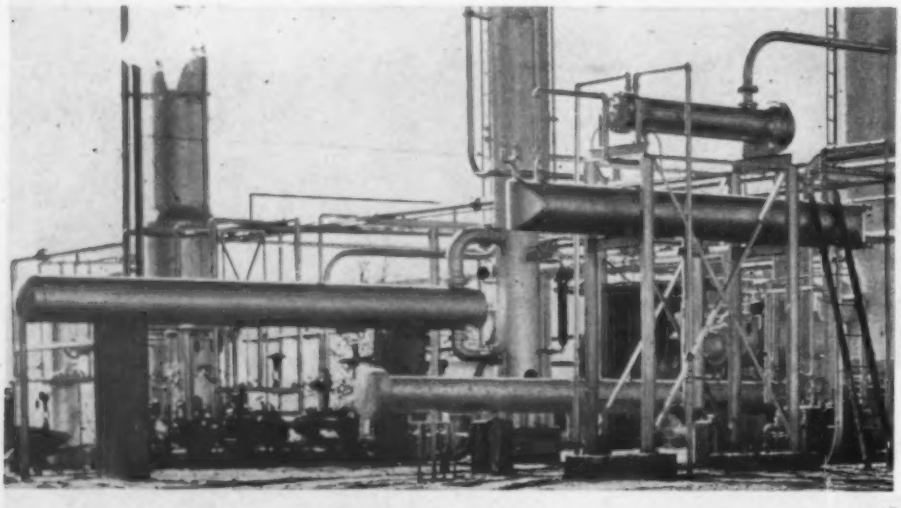


The base of tanks can be given adequate drainage by the use of gravel or crushed rock. Rock of 1- or 1½-inch sizes is spread evenly for a thickness of 6 to 8 inches above grade. The rock is retained in place by a metal band attached to 2-inch pipe stakes driven at 6 to 8-foot intervals.

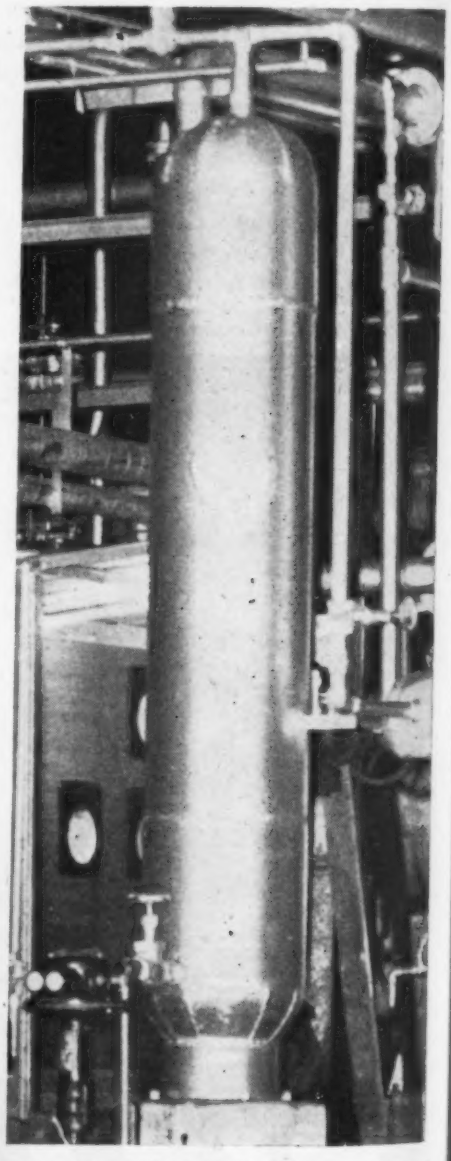
Hose Replaces Manifold To Prevent Contamination

WHEN separating high-purity fractions from a charge to the column in a gasoline plant, the boiling range and composition can be damaged by contamination with other close-cut fractions if a common transfer system is permanently installed. The method used by Skelly Oil Company to prevent mixing consists of separate lines supplemented by high-pressure hose from the accumulator to storage, or from one storage tank to another.

A short length of pipe line is laid from the accumulator, or liquid receiver, and from the various draw-off nozzles on the columns to a practical distance from the equipment. These separate lines rise from the ground and have lubricated



Sections of 23-inch pipe serve as fat-oil flash tank and accumulator.



This small vertical vessel was constructed of 23-inch pipe. It is 10 feet high and filled with a solid adsorbent for dehydrating gas for instrument operation. The adsorbent is regenerated by passing dry gas from the residue scrubber into the top of the dehydrator. Gas enters the bottom and flows to instrument-gas volume tank.

Continental to build small TCC unit at Denver

THIRD SMALL TCC LICENSED SINCE WAR WILL BE FIRST CAT-CRACKER IN COLORADO

As an important part of a $4,000,000 construction program to enlarge its Denver refinery, Continental Oil Company has contracted to build a new small T C C unit of 3,300 B/D fresh feed capacity. Engineering and design of the new unit are already under way and its completion is scheduled for early 1948.

Although the new unit will be the first cat-cracker to be built in Colorado, it will be Continental's fourth T C C, three others of larger capacity having been operating since early 1944 in the company's Ponca City refinery. Improved features of design and construction, which will be incorporated in the new unit, will provide a higher yield of motor fuel than any single catalytic cracking unit in existence.

Houdry will be glad to furnish to any refiner, a complete economic analysis to show exactly what T C C can do for his particular operation.

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plug valves and permanent fittings for connecting hose unions. As many of these lines are laid as products are to be produced so that each fraction will be transferred through its individual system. Additional lines are placed near the product lines but leading to the storage tanks where they are fitted with identical connections and valves as are installed on the product lines.

Each special product tank is equipped with a nipple at the base which acts both as a filling and withdrawal connection. Valves are installed near the tank and extension nipples have permanent fittings for attaching hose.

If rigidly specified blends are to be produced, hose is connected from one tank to another and the particular product, only, is thus transferred without chance of becoming off-specification by accidental entry of another cut, or by the seepage through connections when not tightly shut-off, as may, and has happened when using a permanent manifold system of piping.

Employing a set of twin connections near the fractionation columns, a cut containing two fractions can be charged from one accumulator tank to the surge tank of the following tower without the necessity of going into storage and running the risk of picking up stray fractions laying in lines and in the bottom of storage tanks. Since each line has its number corresponding to a line ending at the storage tanks, and further identified by different colors, the operators need have no difficulty when making connections across with the transfer hose.

Union Oil Company's Manufacturing Department Technical Trainee Program

As Seen by Trainee
WILLIAM K. PARK

THE problem of giving practical training to a newly hired technical college graduate in an industrial organization is not new. The importance of this problem is clear and generally well understood. The consensus among employers shows that technical men hired directly from colleges and placed in industry should be thoroughly indoctrinated in that particular industry. It is at this point that opinions diverge as to a course of action.

This essentially is an economic problem. One method is to assign a man to a specific job in the line organization with a minimum of induction training. A second method involves placing the new man on a general training basis in all phases of work with the aim of equipping him with a much broader and comprehensive knowledge of all departmental operations before assignment to a particular job. This latter program, however, is slower and more costly than the first since full productive effort is not always gained during the training period. Also a somewhat higher turnover of men may be expected.

Union Oil Company of California, at its Los Angeles and Oleum Refineries, has adopted the latter theory of training for young technical men, and has established a well defined program covering most phases of refinery work. This program occupies the first two years employment and is broadly divided into two one-year periods. During the first year, the trainee is assigned to one or two-month periods in the major departments. For the second year, he is placed for two six-month periods in two related departments in which he has shown aptitude and ability during the first year of training. The second year, then, becomes a proving ground in which he is called upon to accept responsibility and perform special duties although not yet permanently assigned.

Periodically, a Committee, composed of the refinery manager, various operating superintendents, representatives from the research and personnel departments, and the manager of employe relations, meet to discuss each trainee's progress and possible permanent assignment.

Following indoctrination in safety and working rules, the trainee is assigned initially to the inspection laboratory to conduct routine tests and to learn how these tests correlate with the control of refinery processes.

From the inspection laboratory the trainee moves to the chemical laboratory where advanced information may be gained. In this assignment, the basic chemistry involved in refining is learned simultaneously with the performance of routine and special tests.

The next move for the trainee involves the radical departure from the routine control laboratories. He is transferred to the Research Department for basic training in the operation of precision measurement instruments. Here also he operates precision small-scale distillation equipment and interprets the results therefrom.

The training program up to this point has had two principal goals. First, the trainee learns how the refinery and its products are controlled by laboratory tests. Second, he develops an appreciation of the analytical tools which will be available to him, and the results which he can expect from these tools. This period builds a foundation for further training on a large-scale refinery equipment.

The next normal assignment places the trainee in the refinery engineering department with the inspection, materials and standards group wherein he gains first-hand contact with operating equipment. By assisting in depreciation surveys, pump and other performance tests, the trainee begins to learn the fundamentals of equipment layout and design. The extent and costs of maintenance work become apparent as well as the safety limits of equipment operation.

The last half of the first year normally is spent in the process department and in one of the operating departments. In the process department detailed studies of several of the refinery units may be made. It is here that the trainee integrates his plant experience and economic education. The true fundamentals of engineering "hows and whys" are taught as the trainee is directed to perform assignments such as plant material balances, economic surveys and recommendations for process improvements.

Upon leaving the process department, the trainee is assigned to one of the refinery operating departments such as distillation, cracking or operating and treating. Such processes as thermal and catalytic cracking, alkylation, fractionation, and product treating are taught by placing the trainee in the process unit along with the plant operators to learn unit operations. During this period the trainee is permitted to take the basic operator training course and to learn fire fighting and the practical operation of such equipment as pumps, prime movers, instruments, etc. Also he comes in contact with scheduling training and other operating personnel problems.

At the beginning of the second year of training, on the basis of recommendations of the "trainee committee" the course of work for the ensuing 12 months is decided upon. After observation and frank discussions between the trainee and the committee representatives, an assignment may be made which is based upon the trainee's preferences and inclinations as well as the foreseen need for technical and supervisory assignments.

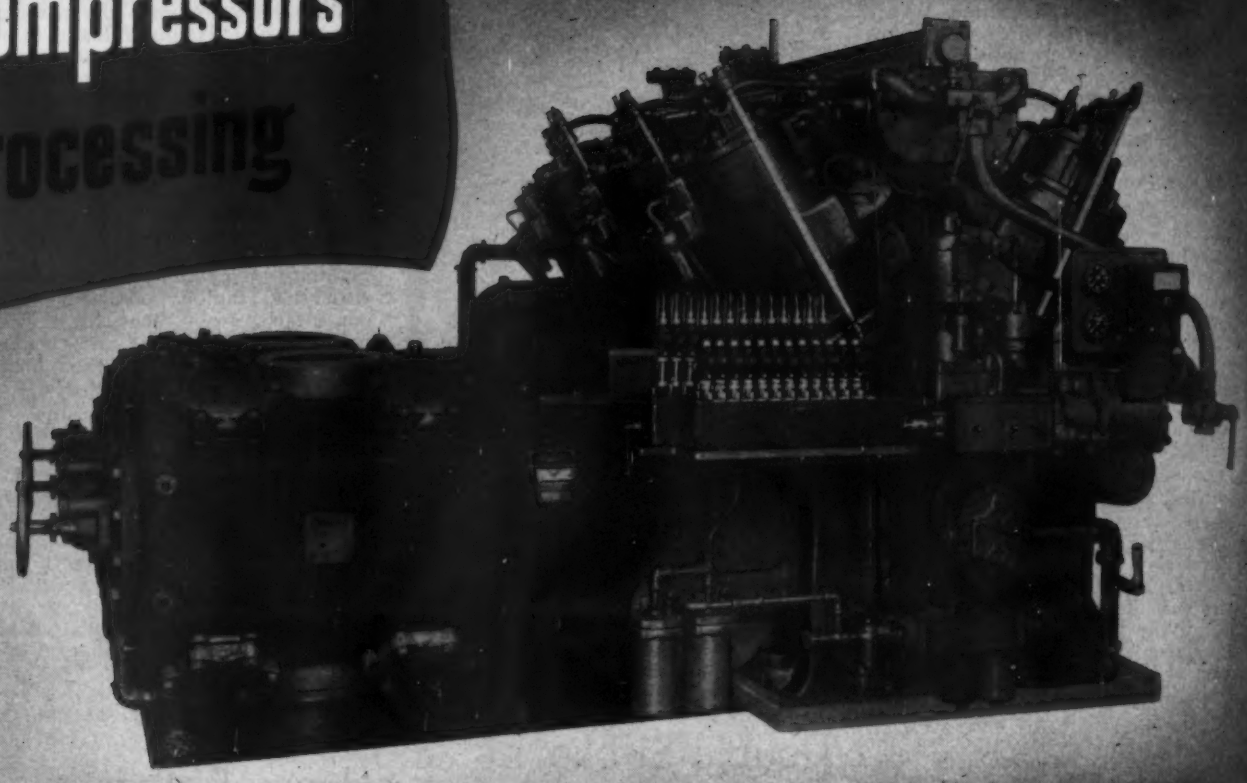
Departments are split generally into maintenance, operations, process engineering, and research groups. The trainee is channeled into one of these broad fields for the second year. During this period he is called upon to accept definite responsibility and to perform considerable productive work. Usually during this period a final evaluation of the trainee's ability and aptitude is obtained.

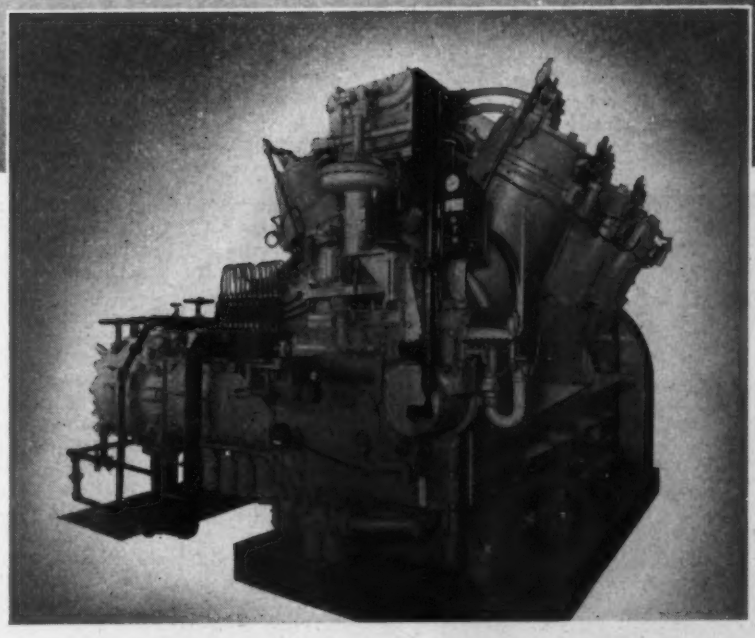
When the two-year training period has elapsed, the trainee is given a final review by the trainee committee for a regular assignment. He has been under the observation of qualified men for a sufficient time to insure placement to the best advantage of both the man and the company. Thus, not only does the training program provide the man with invaluable experience which he might not receive otherwise, but it reduces the chance of error of "the square peg in a round hole."

As by-products of the training program, one may list other important items. By moving the trainee frequently throughout the refinery, he learns how to get along with men which, in later years, will encourage the best interdepartmental and supervisor-employe relations. Second, the trainee learns line organization, job procedures and how recommendations and orders are issued and carried out.

Trainees who have had the benefit of this broad program eventually have an excellent opportunity of advancing into responsible technical-administrative positions and the company has insured itself of constant supply of well-trained technical men.

Compressors processing





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ABOUT OUR AUTHORS . . . . . and Other PERSONALITIES

New CNGA Secretary

In expressing a welcome to E. R. Millett, Jr., now secretary-treasurer of the California Natural Gasoline Association, it is only fair to him to admit the regret that comes with noting the resignation of George L. Tyler. The official announcement, however, is reassuring in a measure in that Tyler is due to remain close to the industry as the representative of the petroleum chemicals division of E. I. du Pont de Nemours & Company.

Millett has devoted most of his work since graduation from Pomona College to consulting work in the petroleum industry. Only recently he was discharged as a captain in the army after service in Japan and Korea. For 10 years previous to the war he was in consulting work. Previously he had been with Union Oil Company for three years and he was for one year on the staff of Martin Van Couvering.

All the while he has been active in civic affairs, as is shown by this part of the news release from CNGA:

"While working as a consultant he also found time to serve a year as president of the Venice Lions' Club, two years as president and five years as a member of the board of directors of the Venice branch of the Los Angeles Chamber of Commerce, as a member of Mayor Bowron's committee investigating the Los Angeles sewage-disposal problem. He is the organizer of the Shoreline Planning Association, a statewide organization whose purpose is the preservation of the state's beaches and the construction of beach highways."

Taylor has been secretary of the California Natural Gasoline Association for nine years "during which time the group has enjoyed steady growth in membership and has received wide recognition for the work it is doing in its particular field," to quote from another paragraph of the news release.

GEORGE L. TYLER

E. R. MILLETT, JR.

Double Stroke

Back in December when a copy of the paper "Chemicals from Petroleum" came along from J. E. Bludworth, its desirability for the April issue of PETROLEUM REFINER prompted inquiry to headquarters of the American Institute of Chemical Engineers, whose publication Chemical Engineering Progress has first call on these papers.

Then came word from F. J. Antwerpen, editor of Chemical Engineering Progress to the effect that the paper had been released for general publication. It was all in ample time for this issue of PETROLEUM REFINER, including an opportunity for proof reading by the author, who is now in consulting work with Corpus Christi as headquarters.

Before all of these details had attention, along came "Utilization of Natural Gas for Chemical Products" from the United States Bureau of Mines. This brought hurried conversation with W. C. Holliman at the Bartlesville Experiment Station, who reported that the original tracings for the illustrations were in the Pittsburgh office.

Again requests resulted in prompt responses, with the result that the tracings were on hand within a week and plates were completed in ample time for April presentation.

Holliman and Harold M. Smith have done a thorough job of expanding the list of chemicals that are being made or promise to be made from natural gas. Duplication with other recent presentations is unavoidable in these papers but this is of minor consideration when the primary object is to keep advised on progress in petro-chemical processing.

Hanlon Award

Although the Hanlon name no longer has corporate connection within the natural-gasoline industry, the personal interest of E. I. Hanlon remains at its former high status. The Hanlon Award is striking evidence of his appreciation of a phase of the industry that has grown to full stature during his association with the petroleum industry.

Furthermore, the shift of Hanlon properties brings other men into consideration as recipients of this award, presented each year as a part of the NGAA program. When originally provided, Hanlon specified that no one connected with the Hanlon companies could be considered. Now the field is widened.

And this seems like an excellent opportunity of listing the nine men who wear watches as evidence of the award and whose names are on the plaque: Richard C. Alden, Phillips Petroleum Company; James W. Cowles, Shell Oil Company; George G. Oberfell, Phillips Petroleum Company; George Granger Brown, University of Michigan; Alois Kremser, Standard Oil Company of California; Edwin O. Bennett, consultant, formerly with Continental Oil Company; Franklin P. Peterson, Mid-Continent Petroleum Corporation; Meinhart H. Kotzebue, Gasoline Plant Construction Corporation; James E. Pew, Sun Oil Company; and William N. Lacey, California Institute of Technology.

Happy Birthday

It will be a few weeks late, still it will be proper to say happy birthday to Bill Warren, Joe LaFortune, H. E. Felt and others of Warren Petroleum Corporation. The three do not have simultaneous birthdays but the company was 25 years old March 15, 1947.

A silver anniversary issue of "The Natural Gasser" gives some facts which are of more significance than the record of any one company. What has taken place in the instance of Warren Petroleum Corporation is typical of the industry.

Bill Warren had $300.00 and one room for an office when he started out "handling nothing but natural gasoline."

Another First

It is somewhat strange to be adding the words "independent operator" after the name of D. E. Buchanan, who also lists some spot in Colorado rather than Tulsa, Oklahoma, when he registers at conventions. Back when the present NGAA was formed at the Natural Gasoline Manufacturers Association, Buchanan was with Chestnut & Smither Corporation. With its dissolution, his company connection became Hanlon-Buchanan, Inc., which only last year became a part of Warren Petroleum Corporation.

Buchanan will preside over the question-and-answer session of the NGAA convention, known as "Information Please." As such he is designated as moderator.

THE MONTH IN THE INDUSTRY

NGAA Program to Stress Role of Light Hydrocarbons in Motor Fuels

One session of the program of the annual meeting of the Natural Gasoline Association of America in Dallas, April 23, 24 and 25, has been set up to give the industry an insight into its place as a source of motor fuel. This will be the program of the afternoon of April 24 in the main ball room of the Baker hotel, and the theme will be motor fuel.

In general the program for the twenty-sixth annual meeting will follow this pattern:

April 23, cycling operation session in the afternoon.

April 24, general sessions morning and afternoon.

April 25, Information Please.

The buffet dinner and entertainment program, sponsored by the Natural Gasoline Supply Men's Association, will be held the evening of April 24.

All activities will be in the Baker hotel.

The two general sessions of April 24 will start with the address of J. H. Dunn, president of the association and president of Shamrock Oil & Gas Corporation, Amarillo. He will discuss the economic position of the products of natural gasoline and cycling plants under the title, "Shortages," and will deal primarily with the reasons for increasing supplies of liquefied petroleum products. Next will be an address, "Public Opinion and the Future of the Oil Business," Jake L. Hamon, Cox & Hamon, Dallas, vice president of American Petroleum Institute.

Relationship of the trend toward higher octane requirements and the conservation of petroleum will be the theme of a paper by E. O. Thompson, Texas Railroad Commission, whose title is "Octane versus Conservation."

The Hanlon Award will be presented at the close of the morning general session. In the afternoon the session "has been designed to reduce the confusion of ideas with respect to future cars and motor fuels" with the following papers:

"Motor Fuel Trends and Desired Characteristics," William S. James, Ford Motor Company.

"Factors Affecting Quality and Supply of Internal - Combustion - Engine Fuels," William M. Holaday, director of research, Socony-Vacuum Oil Company.

"Natural Gasoline Looks Ahead," G. G. Oberfell, vice president, and R. C. Alden, director of research, Phillips Petroleum Company.

Discussion will follow each of these papers.

Committee sessions and registration will get underway the morning of April 23, bringing the first part of the program for that afternoon, when there will be a report of the corrosion research project committee by T. S. Bacon, Lone Star Gas Company, chairman. Other subjects on this cycling program will be:

"Application of Cycling of Gas Injection Below Normal Gas-Water Contact," H. L. Hensley, Humble Oil & Refining Company.

"Mobile Laboratory for Gas Condensate Studies," E. H. Koepf, The Atlantic Refining Company.

"The Nowata Road Tests," K. C. Bottenberg, Phillips Petroleum Company.

The "Information Please" session has been scheduled for the morning of the last day but will be continued into the afternoon. D. E. Buchanan will serve as moderator and will be assisted by a question-screening committee of F. B. Haverfield, Continental Oil Company; Allen T. Givens, Coltex Corporation; Charles W. Miller, Midland Gasoline Company; M. R. Church, Shell Oil Company, and A. H. Weil, United Gas Pipe Line Company.

The panel of experts has been selected to give answers in the several phases of gasoline- and cycling-plant operation, as follows: Plant and field operations, G. W. McCullough, Phillips Petroleum Company; D. N. Barrow, Arkansas Fuel Oil Company, and J. L. Nutter, Hagy, Harrington & Marsh, Processing and Construction, H. M. Nelly, Jr., J. F. Prichard & Company; Refining Operations, Walter W. Scheumann, Cities Service Oil Company; Research, G. G. Brown, University of Michigan; Frank Fisher, Skelly Oil Company, Walter Dayhuff, Standard Oil Company of California; Safety, Henry Boggess, Sinclair Prairie Oil Company; Legal and Regulatory, Binfold Arney, Attorney; J. K. Baumel, Texas Railroad Commission; Turbines and Turbo-Blowers, B. L. Spain, Ingersoll-Rand Company; Liquid Metering, A. J. Kerr, Pittsburgh Equitable Meter Division, Rockwell Manufacturing Company.

Conventions

April
7-10—National Association of Corrosion Engineers, Palmer House, Chicago.
17-18—National Petroleum Association, Cleveland Hotel, Cleveland.
23-25—Natural Gasoline Association of America, Baker Hotel, Dallas.
30- 2—Natural Gas Department, American Gas Association, Stevens Hotel, Chicago.

May
4- 7—Petroleum Equipment Suppliers Association, Mark Hopkins Hotel, San Francisco.
6- 8—Petroleum Industry Electrical Association and Petroleum Electrical Supply Association, Rice Hotel, Houston.
6- 8—Southwest Gas Measurement Short Course, University of Oklahoma, Norman.

June
2- 3—American Petroleum Institute Division of Refining, Midyear Meeting, Jefferson Hotel, St. Louis.
23-25—American Chemical Society, Midwest Regional Meeting, Kansas City, Missouri.

September
15-17—National Butane-Propane Association, Jefferson Hotel, St. Louis.
29- 3—American Gas Association, San Francisco.

October
21-25—Pacific Chemical Exposition, San Francisco.

November
10-13—American Petroleum Institute, Stevens Hotel, Chicago.

Four Technical Sessions For API at St. Louis

Four technical sessions and a dinner meeting will make up the program of the mid-year meeting of the Division of Refining of the American Petroleum Institute, June 2 and 3, Jefferson Hotel, St. Louis.

The morning session June 2 will be for activities of the division and committees will have an opportunity to report on their work. Albert E. Miller, Sinclair Refining Company, is in charge of this program.

In the afternoon there will be a session on design, construction, inspection and repair of refining equipment. Details of its preparation are under the direction of Walter Samans, Sun Oil Company.

The dinner meeting Monday evening will have a program of general interest, under the preparation of Dr. J. Bennett Hill, Sun Oil Company.

Morning and afternoon sessions will be on the general subject of gasolines and "will cover the whole subject and its realtionship to other refinery products. W. M. Holaday, Socony-Vacuum Oil Company, is in charge of the development of this program.

The general committee of the Division of Refining will meet at noon Tuesday. Other committee meetings will be arranged both during the two-day session as well as for dates immediately afterward.

Corrosion Program Includes Oil Industry Symposium

A symposium for the oil industry, the afternoon of April 9, was part of the program for the annual meeting of the National Association of Corrosion Engineers in the Palmer House, Chicago, April 7-10.

Chairman for this symposium was J. M. Pearson, Sun Oil Company, Chester, Pennsylvania, and co-chairmen were W. F. Rogers, Gulf Oil Corporation, Houston, and A. Wachter, Shell Development Company, Emeryville, California.

Four papers were presented on the program:

"Arsenic as a Corrosion Inhibitor in Sulfuric Acid," A. Wachter, R. S. Treseder and M. K. Weber, Shell Development Company, Emeryville.

"Silicates for Corrosion Inhibition in the Oil Industry," William Stericker, Philadelphia Quartz Company, Philadelphia.

"Cathodic Protection of Pipe Lines," Alan C. Nelson, Plantation Pipe Line Company, Atlanta.

"Effect of Carbide Structure on Cor-

rosion Resistance of Steel," R. W. Manuel, Phillips Petroleum Company, Bartlesville.

Thayer, Bates Head Sun Manufacturing Department

Election of Robert G. Dunlop as president of Sun Oil Company, and Clarence H. Thayer as vice president in charge of manufacturing was part of the change in official personnel that followed the annual meeting of stockholders in Philadelphia March 18. J. Howard Pew, who previously had resigned as president, will continue as a director. Joseph N. Pew, who had been executive vice president, was chosen as chairman of the board.

Previously Thayer was chief engineer and assistant to Aruthur E. Pew, Jr., who resigned as vice president but will remain as a director. Dr. John R. Bates was named assistant to the vice president in charge of manufacturing.

Thayer has been with Sun Oil Company since 1926 when he joined the engineering staff of the Marcus Hook plant. He was successively superintendent of pressure stills and superintendent of the plant before being transferred to the home office, where he was designated chief engineer in 1933. He was elected to the board of directors in 1941. Before joining the Sun organization Thayer had been with Standard Oil Company of California at Richmond, where he started as office boy and advanced to assistant superintendent.

Dr. John R. Bates left the faculty of the University of Michigan in 1936 to join the staff of Catalytic Development Company, operating subsidiary of Houdry Process Corporation. Four years later he was promoted to director of research, a position he held until 1942 when he joined the manufacturing department staff of Sun Oil Company. In 1942 he was appointed assistant to the vice president in charge of manufacturing. His undergraduate schooling was at Amherst University, followed by graduate work at Princeton University from which he earned his doctor's degree in 1927. After study abroad he returned to Princeton in 1929 as research associate and remained until 1932 when he went to the University of Michigan.

Bates was one of five new directors added by action of stockholders, others being Frank R. Markley, general sales manager; Donald P. Jones, also elected comptroller; William D. Mason, director of industrial relations, and R. W. Pack, general manager of the Gulf Coast Division, Beaumont.

The post of chief engineer of the manufacturing department went to Paul D. Barton, who has been assistant manager of the engineering division. Barton has been with the company since 1934, although his consulting work brought him into contact with the company earlier. He is a graduate of the University of Oklahoma and California Institute of Technology in 1920. He went with General Petroleum Corporation in research work concerning lubricating oils. Three years later he became superintendent of the natural-gasoline operations for United Oil Company, now a part of Richfield Oil Corporation in the Signal Hill field. Later he became general superintendent of this department and was the first president of the California Natural Gasoline Association, in which he still holds membership. In 1926 he engaged in consulting work and it was in this activity that he came into the activities of Sun Oil Company.

American Gas Association To Meet in Cleveland

The 1947 annual meeting of the American Gas Association will be held in Cleveland, October 6, 7 and 8 instead of in San Francisco, as had previously been arranged. The California city was selected at the close of the 1946 convention. But the executive committee announced the change, since indications are that attendance will tax hotel facilities of San Francisco.

Kirkbride Named Director Of Laboratories for Houdry

C. G. Kirkbride has been named director of laboratories of Houdry Process Corporation, Philadelphia and Marcus Hook, Pennsylvania. Since July, 1944, Kirkbride has been professor of chemical engineering at Texas Agricultural & Mechanical College. During this period he has prepared several articles for PETROLEUM REFINER and served as program chairman for the American Institute of Chemical Engineers.

C. G. KIRKBRIDE

He is a graduate of University of Michigan. Before joining the faculty at A. & M. he was on the research staff of Pan American Refining Corporation, Texas City, and later on the research staff of Magnolia Petroleum Company at Dallas.

CLARENCE H. THAYER

DR. JOHN R. BATES

PAUL D. BARTON

Western Refiners Discuss Processes And Sources for Future Fuel Supply

L. S. DANIELS, Associate Editor

The thirty-fifth annual meeting of Western Petroleum Refiner's Association at San Antonio March 24, 25, and 26, in addition to technical sessions in which various phases of petroleum refining were treated, included discussions of problems of future petroleum and gas supplies.

Technical discussion was initiated with a joint meeting March 24 to hear reports of committees on motor fuel, tractor fuel, and diesel fuel. Paul Williams, the Association's technologist, conducted the program and read the report of the committee on diesel fuel, the other reports being brief and concerned chiefly with sulfur specifications in legislation on motor fuels, and with the burning of cracked fuels in "pot" burners. Work is planned to investigate the question of sulfur contamination of motor fuels from the users standpoint. The committee on diesel fuels reported considerable activity in their field along the lines of substitute fuels for straight-run fractions. It was recommended that the committee should do all that it can to assist in parallel interest work being performed by the CRC Diesel Fuels Division. The latter has recognized two broad objectives: (1) To secure necessary comparative operational data to determine the range of fuel quality variation compatible with the most economical fuel and operating costs, within the limitations that can be tolerated in existing diesel engines, and (2) to secure basic information to assist in ultimately developing the best combination of diesel engines and fuel characteristics, with due consideration being given to first cost, operation, and maintenance.

Motor-Fuel Trends

L. D. Mann, of Cities Service Refining Corporation presided at the morning session March 25. "Motor Fuel Trends in Relation to Antiknock Requirements" by T. H. Risk and Jane F. Jordan, of Ethyl Corporation, was read by Risk, who said that regular grades of gasoline in the 80 Motor, 88 Research octane range and premium grades in the 85 Motor, 95 Research octane range will be generally available in the next few years, and that while present engines are not designed to take full advantage of these fuels, adjustment of engines to realize more fully the available performance will result in engines gradually beginning to require the quality of fuel made available generally in any locality. Variations with engine speed of both engine-fuel requirement and antiknock characteristics is important and should be taken into consideration along with octane requirement of the engines at the speed of maximum knock and the road octane number of the available fuels. Present engines are critical at low speeds with respect to knock, though their requirement generally falls off rapidly with increasing speed. The introduction of the fluid torque converter, however, coupled with increased compression ratio, may give vehicles which have a far more constant requirement over the speed range. Such vehicles will be more critical at high speeds and as critical at low speeds as the cars now in operation. Fuel sensitivity, the difference between octane ratings by the Motor and Research methods, is good protection against low-speed knock, but extremely sensitive gasolines sometimes show rapid decline in road antiknock characteristics with increasing speed and are susceptible to high-speed knock, and the best guarantee of satisfactory performance at all road speeds appears to be high sensitivity coupled with high lead susceptibility.

Hydrocarbon analyses of fuels of this type indicate that they are obtained by concentrating the olefinic components in the lightest fractions and keeping the olefin content of the heavy fractions as low as possible. Knowledge of the relation of these factors to plant operation will help refiners to use existing equipment to best advantage and to make wise decisions as to the types of new processing equipment to install in order to complement their present plant.

(This paper appears at page 99 of this issue).

Catalytic Cracking Processes

A panel discussion of problems encountered in the commercial operation of several catalytic cracking processes was conducted by Ray R. Irwin, Socony-Vacuum Oil Company, and featured a series of questions prepared by process operators which were answered by representatives of process licensors. J. B. Godwin, Magnolia Petroleum Company, prepared questions on the TCC process which were answered by H. D. Noll, Houdry Process Corporation. John W. Bertetti, Root Petroleum Company, prepared questions on the Fluid process which were answered by Donald J. Bergman, Universal Oil Products Company, while C. O. Anderson, Globe Oil & Refining Company, Lemont, Illinois, prepared questions on Cycloversion which were answered by Hugh A. Neal, Phillips Petroleum Company.

Government Representatives Heard

The afternoon of March 25, E. O. Thompson, chairman of the Texas Railroad Commission, delivered an address of welcome, and then discussed problems of gas conservation. A telegram from Secretary of the Interior Krug, read to the meeting, said the National Petroleum Council in no case will make any effort to run the oil industry, and advocated minimum government control, and emphasized the advantage of the government and industry working together.

R. B. Tollett, Cosden Petroleum Corporation, Big Spring, Texas, presided at this session.

"Petroleum and Natural Gas—Uses and Possible Replacements," by R. E. Wilson and J. K. Roberts of Standard Oil Company (Indiana), was presented by Dr. Roberts and described sources of oil, gas, gasoline, and other petroleum products, and for coal as follows: 24 billion barrels of proved petroleum oil reserves, imports from foreign producing areas which are expected to play a part of increasing importance, and synthetic fuels to be produced by synthol type processes which will also provide by-product petroleum chemicals. It was forecast that synthol plants would appear in this country in commercial operation somewhat along this timetable: plants utilizing natural gas as feed stock, 1947; utilizing residual fuel oils, 1965; utilizing coal, 1970.

"Fueling the Navy" was the subject of Captain Ralph E. Wilson, USN, who recounted problems in logistics confronting the Navy in the war, and told of building storage facilities in advanced bases in the Pacific, pipe lines across Panama as protection against possible blocking of the canal by enemy attack, and of pipe lines and fuel supply arrangements used in the European invasion. Captain Wilson stated that petroleum requirements in any future conflict, can be expected to increase at least 30 percent, and emphasized the necessity for cooperation between the military and industry in such an event.

"Hydrocol" Development

G. R. Kinter, Tidewater Associated Oil Company, Tulsa, presided at the technical meeting held the morning of March 26.

P. C. Keith discussed development of the "Hydrocol" process from its starting point in the German work with Fischer-Tropsch processes, and expressed his views of the probable effect of the process on reserves of liquid fuels.

Keith recounted difficulties encountered by the Germans in the operation of the Fischer-Tropsch plants using brown coal as a starting point in the manufacture of gasoline and other synthetic-oil products, and stated that process and economic improvements in catalysts, engineering improvements in synthesis-gas preparation methods, reactor design, waste-heat recovery, and very important improvements in product quality, have resulted in a process which resembles the original only in that similar chemical reactions are involved. The "Hydrocol" process, he said, will give yields of 65 to 80 percent gasoline of 75-80 CFRM octane number, plus a variety of by-product chemicals, as alcohols, ketones, organic acids, and other oxidation products. Plants now building at Brownsville, Texas, and at the Hugoton field in Kansas, will utilize natural gas as a fuel to produce 7000 barrels per day of synthetic crude oil each. The process involves the reaction of carbon monoxide and hydrogen, and these feed materials can be obtained from a variety

of sources, in addition to natural gas, including heavy fuel oils and coal. Plant investments are higher with feed other than natural gas because additional feed preparation equipment is required, but the engineering problems of such plants have been largely worked out, and economic considerations can be expected to control their development. Coal gasification processes used commercially in Europe have thermal efficiencies in the order of 80 percent.

Plans recently announced by the Pittsburgh Consolidation Coal Company call for expenditures in excess of $120,000,000 in the construction of facilities for the gasification of coal. This work, which is expected to be in commercial operation by 1951, primarily will produce fuel gas with gasoline and diesel fuels as by-products. The program is being undertaken in cooperation with Standard Oil Development Company, whose president, Robert P. Russell has stated that his parent company, Standard Oil Company (New Jersey), wants to perfect the new gasification process because it would assure an enormous potential reserve of liquid fuel, and because the cost of finding and producing petroleum oil is mounting and probably will continue to rise. Russell has said that his company is confident that oil reserves will remain as large 20 years from now as they are today, but that coal is by all means the greatest source of energy. Commercial reserves of all grades of coal total about 3.18 trillion tons. If half of this can be used for synthesis, coal could represent a potential gasoline reserve, for example, of 3.7 trillion barrels, "or enough to last for 3000 years."

Natural Gas Not Irreplaceable

Keith asserted that the fuel gases from coal gasification are suitable feed for the "Hydrocol" process, and that such plants will make us independent of natural gas and petroleum reserves as the only source of liquid fuels, that natural gas is not "an irreplaceable natural resource." The "Hydrocol" process was also suggested as being of utility to petroleum refiners in realizing maximum yields from crude oils. He explained that little investigation along these lines has been completed, but that preliminary examinations indicate the economic utility of the process as a "scavenger" for all low-value refinery hydrocarbons. A refiner on Mid-Continent crudes, for example, might well charge light gases usually required for refinery fuel to a "Hydrocol" unit while using residual fuel oil to supply plant heat. This fuel oil would have to meet no specifications other than the requirements of combustion in the refiner's own furnaces, and the light gasoil normally required to "cut-back" residual stocks to a marketable fuel oil product could be diverted to more profitable uses. This refiner would thus have no residual fuels to market and often would be greatly benefited thereby. Another case mentioned applied to the refiner with crude supplies heavy in asphaltic materials resulting in the production of large percentages of residual fuel oils. This refiner might do well to charge his fuel oil production, rather than the plant fuel gas, to a "Hydrocol" unit, since the route followed in the earlier case would leave an excess of black fuel oil over plant-heat requirements, and this material would still present a marketing problem. With the fuel oil utilized as charge for a "Hydrocol" unit, the light fuel gases in the plant would be used to supply processing heat. Either route has the advantage of increasing the overall refinery gasoline yield compared to other processing schemes, and also markedly improves the overall plant octane since gasoline produced in the "Hydrocol" process is of high octane level.

"Chemical Phase"

The field of petro-chemicals as seen from the viewpoint of the refiner of petroleum who has as yet no experience along these lines was the subject of a paper by M. B. Chittick, The Pure Oil Company. It was stated that the petroleum industry has passed through two phases and has now entered a third and permanent phase. The first phase might be termed the burning oil phase, the second the automobile phase, and the third, which involves the use of petroleum as raw material for chemical manufacture or synthesis, may be termed the chemical phase. Petroleum chemicals were defined as "those products obtained directly or indirectly from crude oil or natural gas and used in the chemical-consuming industries. All those products derived from petroleum and not employed as fuels or lubricants may be classified as petro-chemicals. From recent surveys of all plants under construction for the manufacture of chemicals using petroleum or natural gas as a source of raw material, it appears unlikely that the 1945 totals will seem very large ten years hence."

"From the standpoint of the independent refiner, the simplest and safest approach would appear to start with a re-examination of his particular crude, or crudes, with the objective of determining what petro-chemicals are components of that crude and whether or not they can be extracted and/or refined with existing facilities or by the addition of new facilities involving a minimum capital outlay. When these possibilities are determined, an exhaustive market survey should be conducted to determine the overall economics of the proposed operation." A number of product possibilities based on separation and purification of the components of various crudes were enumerated.

Chittick continued, "The second approach to the petro-chemical field is, perforce, on a broader and more elaborate scale and has to do with the synthesis of petro-chemicals from raw materials which are components of natural gas, crude oil, or obtained as by-products in normal refinery operations. Before any decision can be made intelligently as to the advisability of entering the field on this more elaborate scale, the following preliminaries should be followed: a thorough search of the literature, a thorough patent search, and a thorough market survey."

Many difficulties inherent in the placing of a new product into commercial production were pointed out, and economic factors of supply and demand were discussed. Chittick concluded: "Figuratively speaking, the surface of the petro-chemical industry has only been scratched. It must and will expand." It is believed that the future of the industry can be viewed with optimism.

Future Supply and Demand

R. W. McDowell of the Mid-Continent Petroleum Corporation, was the presiding officer at the general session March 26. S. A. Swensrud, The Standard Oil Company (Ohio), presented a discussion of "Various Factors in the Future Supply and Demand Picture" in which it was stated that much of the past work in the prediction of demand for petroleum products has been on the low side, in spite of careful studies and reasonable assumptions. A qualified industry committee in 1925 forecast that by 1945 total crude oil runs to stills would be 3.8 million barrels daily, and a similar authoritative study in 1935 forecast 3.0 million daily. Runs in 1945 actualy were above 4.8 million barrels daily. The forecasters in both cases foresaw more motor vehicles but understandably failed to foresee that motor vehicles would cover much more mileage per year as roads were improved and that they would utilize better engine efficiency mainly for achieving better performance and comfort rather than for cutting down on weight of cars or increasing mileage per gallon. Consequently, gasoline use has far exceeded those earlier forecasts. The early forecasters left out a consideration which has entered the picture and proved important, stated Swensrud. They base their estimates on gasoline as the primary and ruling product from crude oil. Actually, heating oil, tractor fuel, and other products have assumed great importance and have much weight in determining the volumes of runs to stills. So strong has been the demand for these other products that in order to meet it, the industry has deliberately processed enough crude to obtain those products even though in doing so, gasoline yields had to be held down to avoid surplus production of the main product.

While much petroleum was used during the war, the use was not abnormal but only in line with the prewar and postwar tendency to increase constantly.

Other recent estimates of demand also have proved too low, several by 300,000 barrels daily for early 1947. On the conservative side, for example, were recent estimates of both the Interstate Oil Compact Commission and the Independent Petroleum Association of America.

"I am inclined toward relatively high volume expectations for the next several years," Swensrud continued. "Until a depression occurs the trend of car (and gasoline) use will continue strong," and a similar outlook for domestic heating oil is foreseen. Other factors to be considered include diesel-oil requirements of locomotives formerly using coal, asphalt requirements for paving which are expected to be great, increasing use of petroleum products in aviation, and the growing use of propane, butane, and other liquefied petroleum products. Swensrud concluded, "over the next several years (barring a depression or the development of exceptionally high prices for our products), the total demand for all petroleum products will exceed 5.5 million barrels per day, and that we will not have reached the end of the road then, either."

"The Rural Market in the Petroleum Industry" was the subject presented by Don Ross, representing the Agricultural Publishers Association. Ross discussed the use of machinery and of petroleum products in modern farming, and offered

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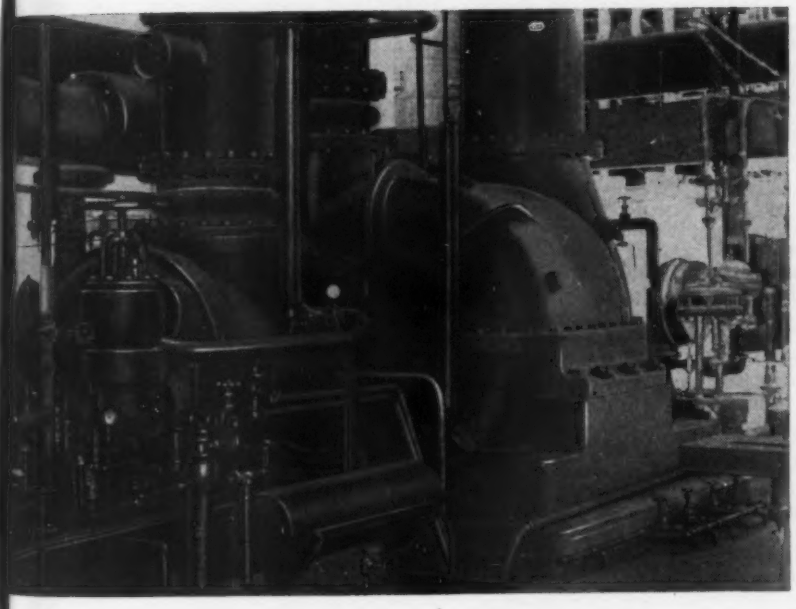

IN WHAT IS PERHAPS the largest single fluid catalytic cracking plant in the world, six Allis-Chalmers turbo blowers are supplying air at the rate of 135,000 cubic feet per minute. Each unit is rated 22,500 cfm and is driven by a 1730 hp condensing steam turbine at 5100 rpm.

For over 50 years, the A-C nameplate has meant highest quality in high speed rotating equipment of many kinds . . . and through the years Allis-Chalmers has furnished almost every type of blower used in refinery processes. Today, Allis-Chalmers offers a complete range of single and multi-stage blowers and compressors.

CHECK LIST OF ALLIS-CHALMERS EQUIPMENT FOR THE OIL INDUSTRY

BELOW, is a partial listing of the equipment Allis-Chalmers builds for Petroleum Refineries—Field and Transportation.

Additional information on any of this equipment is available upon request.

☑ **POWER GENERATION**

Condensing and non-condensing steam turbines from 10 hp to 10,000 kw and up.
Gas turbines to 15,000 hp

☑ **POWER DISTRIBUTION AND CONTROL**

High and low voltage switchgear
Indoor and outdoor circuit breakers
Voltage regulators
Dry type, oil filled and non-inflammable liquid filled transformers
Load center, Multi-circuit and Single circuit unit substations

☑ **CONVERSION EQUIPMENT**

Motor generator sets
Mercury arc rectifiers

☑ **MOTORS AND DRIVES**

Alternating current motors
Squirrel cage and wound rotor
Explosion-proof
Totally enclosed

☑ **TEXROPE DRIVES**

V-Belts
Constant speed sheaves
Variable speed sheaves
Magic grip sheaves

☑ **PRODUCTION, PROCESSING AND MAINTENANCE EQUIPMENT**

Tractors
Pressure vessels and plate work
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evidence to demonstrate the progressive nature and buying power of this market.

Ralph C. Champlin, Ethyl Corporation, gave an address titled "One Hundred Years of Public Relations in Oil" in which he discussed events in the history of the industry in relation to their effect on public opinion, and reported on the progress of the API public-relations program. It was suggested that each individual in the industry could, by making a conscious effort to inform his immediate associates and neighbors on the nature of his work and his company's general activities, perform an extremely valuable service in furthering the general public-relations program.

Oppose Co-Ops

In the first meeting of the newly re-elected directors March 26, a resolution was passed "in opposition of any tax inequalities favoring co-operatives over other types of business enterprise." The secretary was instructed to forward copies of the resolution to the Secretary of the Treasury, the Commissioner of Internal Revenue, the Chairman of the Senate Finance Committee, and the Chairman of the House Ways and Means Committee, together with a request "for such legislative action as may be found necessary to accomplish true tax equality among business enterprises," and officers and counsel of the association were requested "to present the association's position on the relative taxation of co-operatives and regular business corporations to such committees of Congress as may afford public hearings on this subject."

All Officials Re-elected

Officers and directors who served for 1946-1947 were re-elected for the year 1947-1948 for each post subject to voting in the election, as follows:

H. T. Ashton, Socony-Vacuum Oil Company, St. Louis, president; and T. A. Helling, El Dorado Refining Company, Eldorado, Kansas; R. W. McDowell, Mid-Continent Petroleum Corporation, Tulsa; T. M. Martin, Lion Oil Company, El Dorado, Arkansas; and Raymond L. Tollett, Cosden Petroleum Corporation, Big Spring, Texas, vice presidents. These officers were re-elected as directors, as were Paul G. Blazer, Ashland Oil & Refining Company, Ashland, Kentucky; Reid Brazell, Leonard Refineries, Inc., Alma, Michigan; H. W. Camp, Cities Service Oil Company, Bartlesville; Sheldon Clark, Sinclair Refining Company, Chicago; O. L. Cordell, Bareco Oil Company, Tulsa; Frank H. Dunn, Wilcox Oil Company, Tulsa; A. M. Hughes, Phillips Petroleum Company, Bartlesville; Ray R. Irwin, Socony-Vacuum Oil Company, Kansas City; R. E. Luton, The Ohio Oil Company, Robinson, Illinois; F. L. Martin, Sunray Oil Corporation, Tulsa; W. E. Moody, Deep Rock Oil Corporation, Cushing, Oklahoma; I. A. O'Shaughnessy, Globe Oil & Refining Company, St. Paul, Minnesota; L. H. Prichard, Anderson-Prichard Oil Corporation, Oklahoma City; Roland V. Rodman, Bay Petroleum Corporation, Denver; W. F. Sims, Panhandle Refining Company, Wichita Falls; J. W. Vaiden, Skelly Oil Company, Tulsa; W. W. Vandeveer, Allied Oil Company, Cleveland; J. W. Warner, Tidewater Associated Oil Company, Tulsa; C. B. Watson, The Pure Oil Company, Chicago, and Rex H. Winget, Cushing Refining Division, Midland Co-op Wholesale.

Socony-Vacuum Makes Operating Personnel Shifts

Promotions in the operating personnel of Socony-Vacuum Oil Company have been announced following transfer of Howard Sheldon from management of the Paulsboro refinery to New York headquarters, where he has been named assistant director of Socony-Vacuum laboratories. Rea R. Jackson, general superintendent of the plant of Magnolia Petroleum Company, Beaumont, has been transferred to Paulsboro as general manager. At Beaumont, J. L. Heath has been promoted from assistant general superintendent to general superintendent, and C. M. Fabian has been named assistant superintendent.

Sheldon became a refinery engineer at Paulsboro in 1926 and three years later was transferred to New York as chemical engineer, and in time became chief chemical engineer of the research and development department. In 1933 he returned to Paulsboro as chemical engineer and a year later was promoted to assistant superintendent. He became superintendent in 1943 and later that year was named general manager.

Jackson had been with Magnolia Petroleum Company since his graduation from the University of Texas in 1923. He served first as a chemist, later as a stillman and then foreman and as supervisor of processing in operations. He became general superintendent in 1936.

Heath has been at the Beaumont plant since 1915 and has served in various capacities. In 1938 he was placed in direct charge of refining operations as process supervisor. He was named assistant general superintendent in 1944.

Fabian came to the Magnolia plant at Beaumont following graduation from Texas A. & M. College and service in World War I in 1919. His duties have included chemist, process foreman and finally process supervisor in 1944.

Brown Becomes Secretary National Petroleum Council

James V. Brown has been selected to fill the post of secretary-treasurer of the National Petroleum Council, Washington. For several years he has been in the Washington office of the Independent Petroleum Association of America as statistician and analyst. His other activities during nearly 20 years of activity in the petroleum industry include secretary of the Petroleum Industry War Council's production committee, and vice chairman of the economics advisory committee of the Interstate Oil Compact Commission.

Phillips Announces "Nearer Natural" Synthetic Rubber

Phillips Petroleum Company, Bartlesville, Oklahoma, has announced an improvement in butadiene-styrene chemical rubber, which "according to laboratory tests appears to be the equivalent to natural rubber in many important properties." The improvement is credited to the accomplishment of carrying out polymerization at extremely low temperatures. Further details are given in this part of the announcement:

"Although the final answer will come only after gruelling testing of the tires from this new rubber, it appears that the chemists' dream of the synthesis of a man-made rubber equal to, or better than, natural rubber for all sizes of tires is near realization. Although it has been recognized for some time that improvement in synthetic rubber is to be expected from manufacturing processes carried out at very low temperatures, the practical attainment of that improvement has only recently been realized through the development of extremely rapid emulsion polymerization recipies for the production of rubber at such reduced temperatures."

Texas Company Reveals Refinery Expansion Plans

Plans for additional facilities, principally for improvement of product quality, and also for some additional increase in capacities, were discussed in The Texas Company's 1946 report to stockholders. The expansion and modernization program contemplates the following installations:

An East Coast refinery on the Delaware River, near Camden, New Jersey. This refinery is being designed for processing of both domestic and foreign crudes. If materials are available, construction will start during 1947. Crude-charge capacity is planned to be 40,000 barrels per day.

New lubrication oil equipment at Port

Trends of Operations and Changes in Stocks

Figures on crude stocks are from Bureau of Mines weekly reports; all others from American Petroleum Institute weekly reports, which are estimates on Bureau of Mines basis.

(All figures in thousands of barrels—add 000)

Trends in Week Ended	Crude Oil			Gasoline		Gasoil and Distillate		Residual Fuel	
	Production Daily	Runs to Stills Daily	Stocks Week End	Production Weekly	Stocks Week End	Production Weekly	Stocks Week End	Production Weekly	Stocks Week End
1946:									
January 26	4,626	4,553	220,544	13,622	101,737	5,720	29,498	8,411	39,722
March 2	4,726	4,779	229,430	13,871	104,462	5,888	25,148	8,634	38,441
March 30	4,425	4,684	224,994	13,896	104,715	5,337	28,240	8,738	37,746
April 27	4,672	4,685	224,443	14,228	99,631	5,568	30,466	9,204	39,404
May 25	4,759	4,857	222,214	14,322	95,769	5,463	32,973	8,908	43,368
June 29	4,957	4,854	223,883	14,500	92,333	5,325	37,762	8,828	46,447
July 27	4,926	4,896	223,756	14,535	88,626	5,817	44,316	8,217	49,517
August 24	4,836	4,866	225,672	14,639	86,251	5,649	51,405	8,126	52,061
September 28	4,778	4,829	221,903	14,538	85,854	5,450	57,903	8,158	56,914
October 26	4,730	4,758	221,184	14,863	86,423	5,710	65,499	7,728	60,872
November 30	4,795	4,707	225,119	15,145	88,371	5,228	66,062	7,672	58,647
December 28	4,713	4,968	226,111	15,604	93,126	5,931	58,941	8,181	53,427
1947:									
January 25	4,672	4,820	221,655	14,624	99,801	5,630	50,357	8,224	48,558
February 22	4,786	4,860	224,560	14,668	103,904	5,929	40,739	8,542	44,919
March 29	4,865	4,843	225,720	14,396	107,576	5,969	32,737	8,668	43,364

THE MONTH • • •

Arthur refinery, including a vacuum pipe still unit, an additional furfural refining unit, and an additional solvent dewaxing unit which will substantially increase lubricating oil capacity of that refinery.

Plant additions are either under construction or contemplated, at the company's Casper, Wyoming, refinery, at the West Tulsa, Oklahoma, refinery, and at the Lawrenceville, Illinois, refinery. The program includes crude units, fluid catalytic cracking units, and necessary auxiliaries, and is mainly for the purpose of improving quality of manufactured products.

Indiana Makes Research Department Promotions

John A. Bolt has been appointed assistant superintendent of technical services, research department, Standard Oil Company (Indiana), Whiting. He has been in the research laboratories since 1930, when he joined the staff as a chemist. Since 1944 he has been a group leader. Bolt did his undergraduate work at Calvin college, Grand Rapids, after which he did graduate work at the University of Michigan, where he earned a master's degree in chemistry.

Four group leaders recently have been appointed in the research department at Whiting: David M. Bransky, D. E. Bruce, T. B. Tom and Lindsay M. Hobbs.

John E. Swearingen, who has been a group leader at Whiting, has been transferred to the staff of Stanolind Oil & Gas Company, Tulsa. He was succeeded by Carl W. Peters.

Evans Named Director Shell Laboratories

Dr. Theodore W. Evans has been appointed manager of the Emeryville, California, research laboratories of Shell Development Company. He succeeds Walter J. Hund who died February 17.

DR. T. W. EVANS

Dr. Evans joined the Emeryville staff as a research chemist in 1930. Previously he had attended College of Puget Sound in Tacoma and completed graduate work in chemistry at the University of Washington where he was awarded Ph. D. He was active in the early development of alcohols, ethers, and esters from refinery gases, and in development of the solvent extraction theory. Before the war he was pioneering in development of synthetic glycerin from petroleum. By 1941 he was supervising all exploratory studies on new chemicals as well as research on their application in such fields as surface coatings and resins. He was appointed an associate director of research in 1942.

Humble Gasoline Plant At Clear Lake Operating

The Clear Lake gasoline plant of Humble Oil & Refining Company recently started processing 50,000,000 cubic feet of gas daily. A major project in the company's gas conservation program for the Gulf Coast areas of Texas and Louisiana, the plant will produce 36,000 gallons daily of liquid hydrocarbons.

About 31,000,000 cubic feet of the total gas processed is casinghead gas from the Dickinson, Gillock, East Clear Lake, and Friendswood fields that was formerly flared. The remaining 19,000,000 cubic feet is from gas wells in the Dickinson and Halls Bayou fields. Before completion of the Clear Lake plant, this gas was produced directly into the company's Tomball Natural Gas System lines. Approximately 55 gas flares were eliminated when the plant started initial operation.

Supplementing the Clear Lake plant proper is an 1800-horsepower compressor plant built in the Dickinson field to boost to line pressure the oil-well gas from the Dickinson and Gillock fields.

Other Projects

A compressor station of 1600 horsepower was added in the North Crowley, Louisiana, field to make delivery of 8,000,000 cubic feet daily of dry gas from the field into transmission lines. The project completed in April. Compressors were added to the Lovell Lake plant to facilitate gathering and processing of an additional 4,500,000 cubic feet daily of gas, and to boost the pressure so that the gas can be delivered into a transmission line to the field. A 600 horsepower compressor was added to the Amelia field compressor station to inject casinghead gas from oil wells back into the ground at 2,600 pounds pressure. Nearly 2,500,000 cubic feet of gas, formerly flared has been returned to the formation since December 1.

Other casinghead conservation projects built by the company since the war are the Hawkins project (East Texas),

Humble Oil & Refining Company's Clear Lake gasoline plant which on March 14 started processing 50 million cubic feet of previously flared gas. This is one of several units in the company's program of economically conserving casinghead gas in Texas.

THE MONTH • • •

Red Fish Reef, Texas, Chalkey, Louisiana, and Roanoke, Louisiana.

Among the major projects built or under construction by the company is included a low-pressure system to process 8,000,000 cubic feet daily of casinghead gas at the Katy Cycling Plant; a casinghead gasoline plant at Anahuac to process between 35,000,000 and 40,000,000 cubic feet of gas daily and recovery of 30,000 gallons of gasoline, which will be completed by mid-summer at a cost of about $2,500,000, and a joint construction project by Humble Oil & Refining Company and Quintana Gas Company of a gasoline plant in the Tom O'Connor oil field near Refugio to process 35,000,000 cubic feet daily of casinghead gas to produce 36,000 gallons of liquid hydrocarbons when it begins operation late this year.

Shell Building Vacuum Flashing Unit at Wood River

Construction has been started on a vacuum flashing unit which will process feed to the twin fluid catalyst cracking units of Shell Oil Company at Wood River, Illinois. Overhead from topping units will be separated in this new unit of 35,000 barrels daily capacity. Details on the new unit were given in a recent news release as follows:

"The flash tower, whose spherical construction makes it the most unusual feature of the unit, will have an inside diameter of 30 feet. Heat exchangers will contain approximately 50 miles of tubes. In addition, 1½ miles of 6-inch tubes will be built into two vaporizing type furnaces. A transfer line, carrying oil from the furnaces to the flash tower, will have an inside diameter of 4 feet, and a mixture of vapor and liquid will flow through it at a velocity of 280 feet per second.

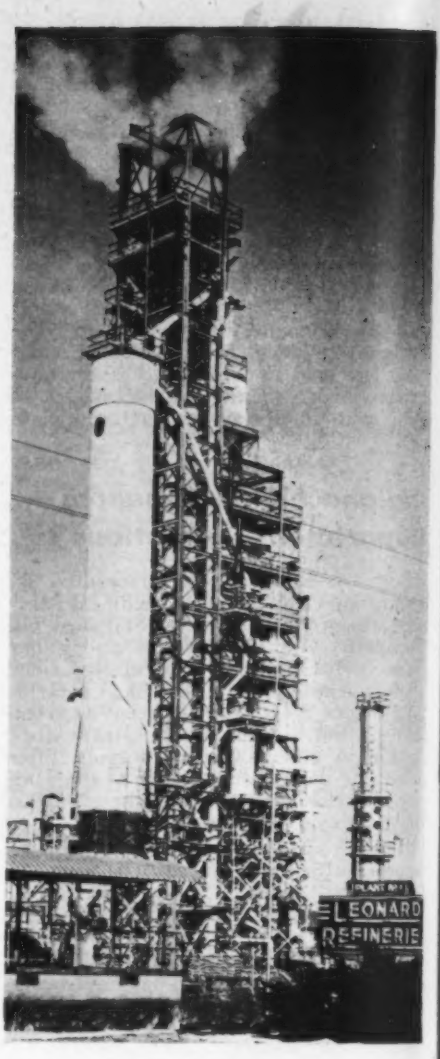
"Operating at 770° F. and 60 millimeters, or 1.16 psia, the new unit will flash 80 percent of the topped crude overhead. Extensive heat economy will be achieved by the use of heat exchange between the products and the crude feed, with over half of the heat tranfer to the oil being accomplished in the unit. After passing through the heat exchangers, the feed will flow through the furnaces, and then through the transfer line to the bottom of the flash vessel. Here, it will enter a cyclone separator which will separate the vapor and liquid.

"Since ordinary steel would be subject to severe corrosion in such a unit, all lines and vessels which will be exposed to oil temperatures of 500° F. or over will be made of chrome molybdenum alloys."

C. F. Braun Company holds the contract for engineering and construction.

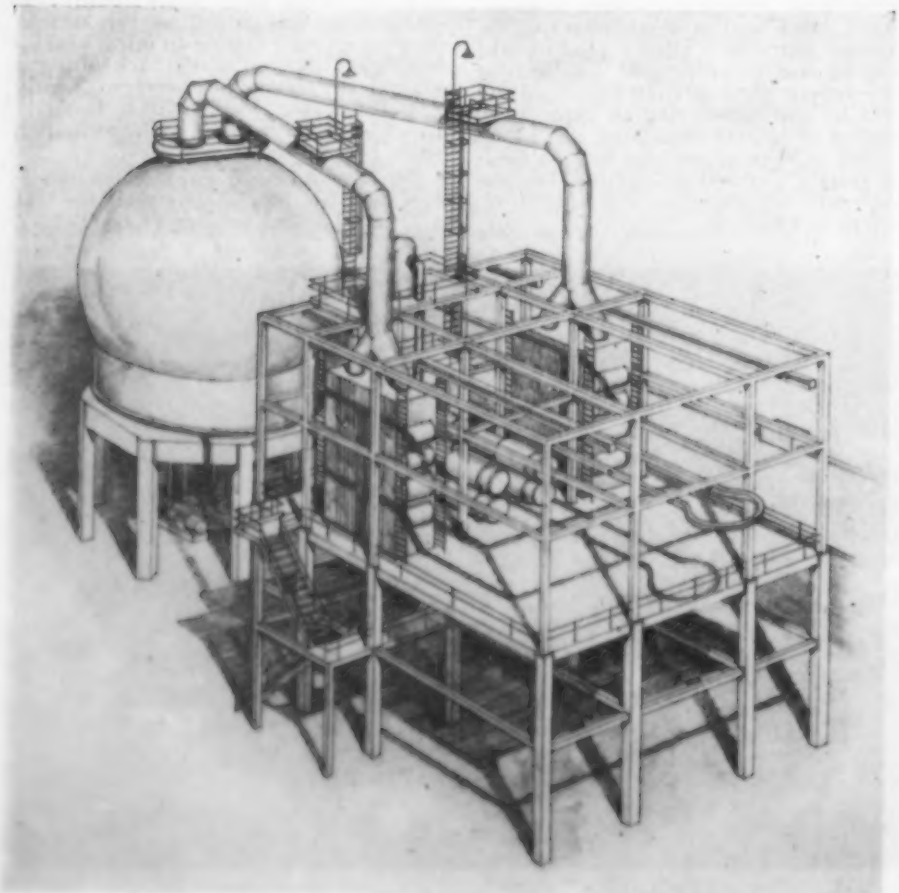
Michigan Refinery Starts Operating Small TCC Unit

Leonard Refineries, Inc., has announced completion at Alma, Michigan, of a new catalytic-cracking unit of the moving-bed type. This Thermofor unit has a design capacity of 3000 barrels per day and was especially designed for small-refinery operations. The unit incorporates several improvements and simplifications, although the plant is similar in its principal design features to larger TCC installations.



Recently completed TCC unit at Alma, Michigan, plant of Leonard Refineries, Inc.

The new Leonard unit is compact in design, with the overall height substantially reduced and the structure considerably simplified. One important simplification is an integral elevator design requiring only one elevator for both spent and regenerated catalyst. Utilization of certain existing thermal equipment in converting the refinery from thermal to catalytic cracking provided substantial savings.



Sketch of vacuum flashing unit being built by Shell Oil Company at Wood River refinery.

Shell's TXL Plant Under Construction

Shell Oil Company is constructing a gasoline plant in the TXL field of Ector County, West Texas, to process 40,000,000 cubic feet daily of casinghead gas. The plant is located a mile east of the new townsite of Notrees, and will obtain gas from the TXL and Wheeler Ranch fields. Residue gas has been contracted for by El Paso Natural Gas Company. Construction of the plant began in December, with completion expected near the end of this year. Previously, gas in the fields has been flared.

Eight 800-horsepower compressors will be used to compress the gas for processing. Electrical power will be supplied by three generators driven by 800-horsepower gas engines. Storage facilities will handle 27,500 barrels.

Clyde B. Gray will be plant superin-

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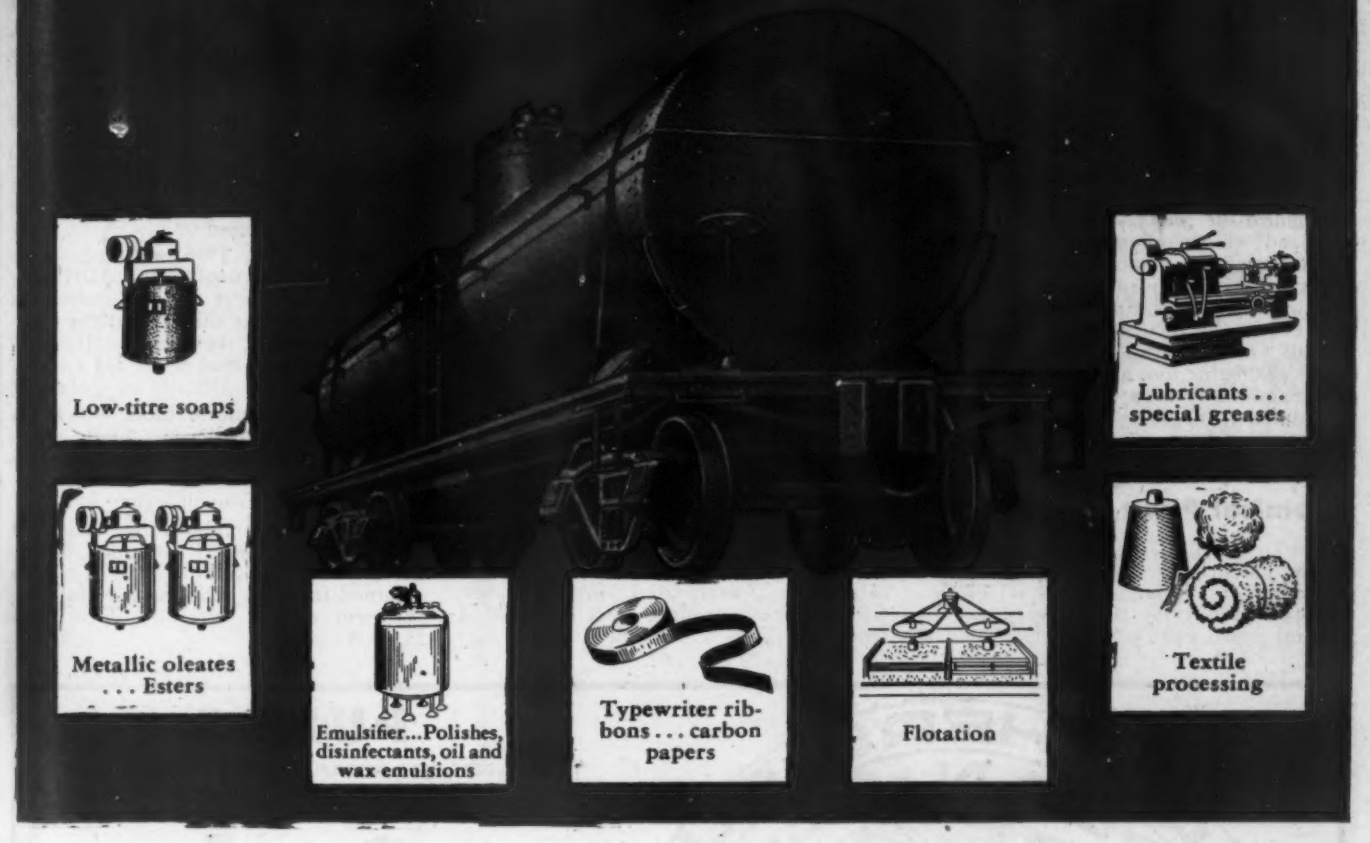
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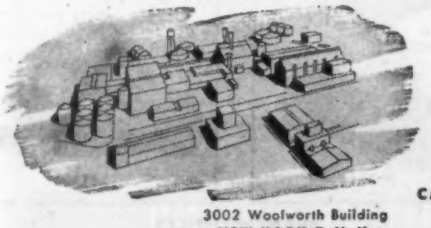

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THE MONTH • • •

tendent, and Frank R. Lovering, supervisor of production in the fields. Operating personnel will be housed in 30 4-room houses in the plant camp, which will also include an office building and a combination dormitory and recreation hall.

Two More Gasoline Plants Planned for West Texas

Two additional gasoline plants have been authorized for West Texas. Sid W. Richardson and associates will erect a plant in the Keystone field, and Gulf Oil Corporation and others will provide the initial plant for Sand Hills field in Crane County.

Richardson and associates have authorized construction of a 70,000,000 cubic foot daily capacity absorption unit to process casinghead gas. Residue will go to El Paso Natural Gas Company and eventually to the California market.

Gulf Oil Corporation has projected a 23,000,000 cubic foot absorption plant for the Sand Hills area, with construction scheduled to begin in June.

Shell Expanding Houston Chemical Plant

Shell Chemical Corporation is currently embarked on an expansion program for its Houston plant which includes facilities for the manufacture of several additional chemicals from petroleum hydrocarbons, according to statements made by J. Oostermeyer, president, and W. P. Gage, vice president in charge of manufacturing, who were in Houston the week of March 3 for a semi-annual technical meeting attended by company engineers and technologists. New process units will be added for the production of synthetic glycerin, ethyl alcohol, isopropyl alcohol, acetone, methyl isobutyl ketone, and methyl isobutyl carbinol. Also subject to further development and expansion in manufacture and application is a soil fumigant, DD, dicholorpropane - dichloropropene, which is expected to find a large market in increasing the yield-per-acre of truck crops.

F. J. Smith Heads Chemical Department of Pan American

F. J. Smith has been named manager of the newly established chemicals department of Pan American Refining Company, and with his assistant, J. F. Scott, has been moved from the company's Texas City refinery to New York. W. T. Herget has been appointed a department head in the Texas City plant and will be responsible for chemical manufacturing activities.

The new department will manufacture petroleum chemicals which have been and will be developed through a program being carried on in the company's Texas City research laboratory. These products will include aromatic solvents, plasticizers, resins, polymers, and miscellaneous unsaturated intermediates. Sales of these products will be handled from the New York office.

Lead Restrictions Lower Gasoline Quality Slightly

Restrictions on the use of tetraethyl lead were reflected in the motor gasolines sold during the summer of 1946, but the quality of the products continued to be high with respect to octane ratings, the Bureau of Mines reported in a semi-annual survey of motor fuels.

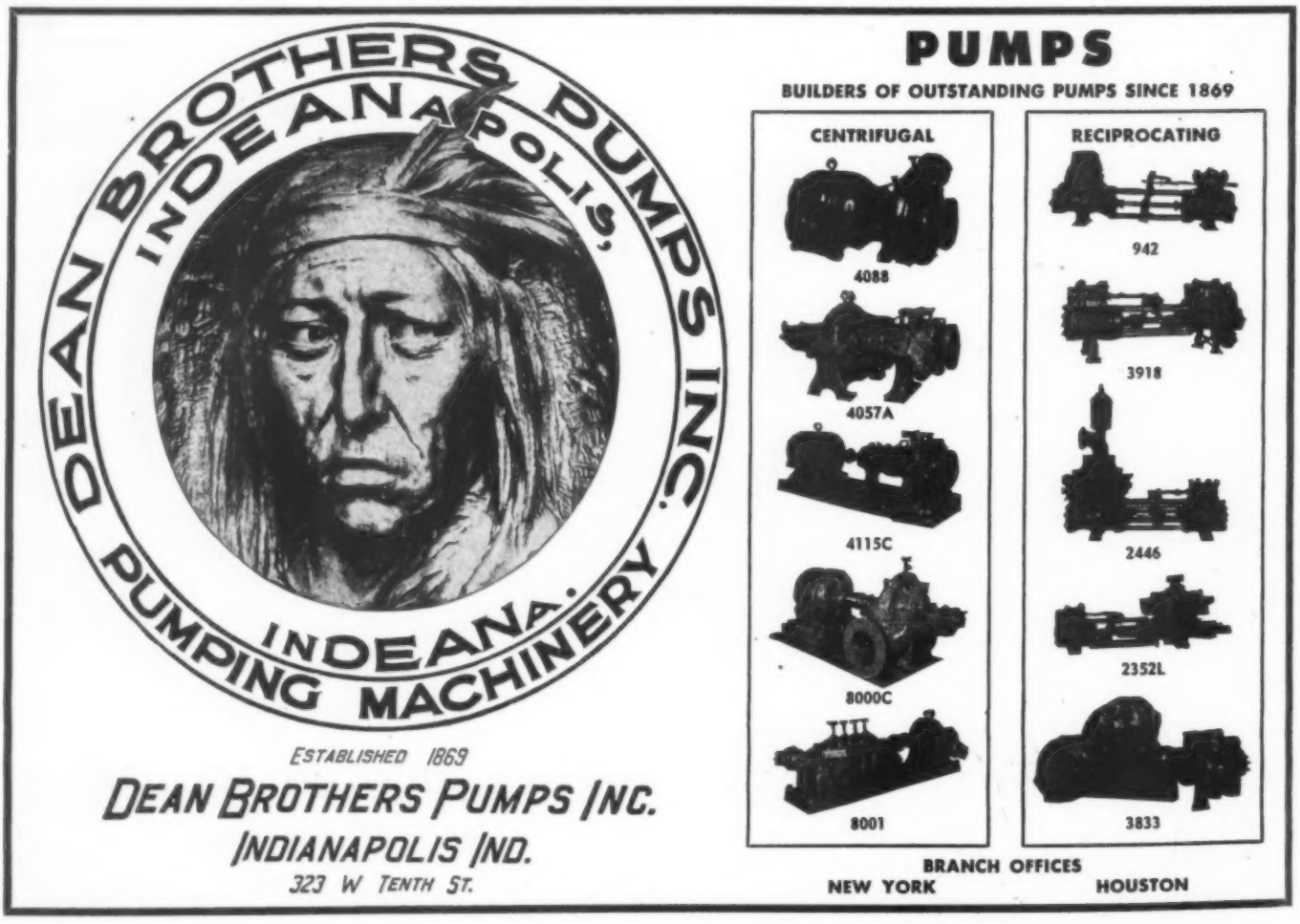
The survey showed that the average octane number of "premium" motor fuels sold last summer was 78.3, compared with 80.9 during the winter of 1945-46, and the average octane number of "regular" gasoline last summer was 74.4, compared with 75.9 the previous winter.

Although the average ratings for the summer of 1946 were below the 1945-46 winter averages, they showed an improvement over the summer of 1945 when premium gasoline averaged 74.9 and regular averaged 69.7.

Results of the 1946 summer survey are based on the analyses of 3,011 samples collected from service stations in 21 marketing areas throughout the country. The samples, representing 113 companies, were obtained from 233 cities in 40 states and the District of Columbia.

The survey of motor fuels, which was started in 1936, again was made in cooperation with the Coordinating Fuel Research Committee of the Coordinating Research Council, Inc.

A copy of the publication, Report of Investigations 4063, "National Motor-Gasoline Survey, Summer 1946," may be obtained from the Bureau of Mines, Department of the Interior, Washington 25, D. C.



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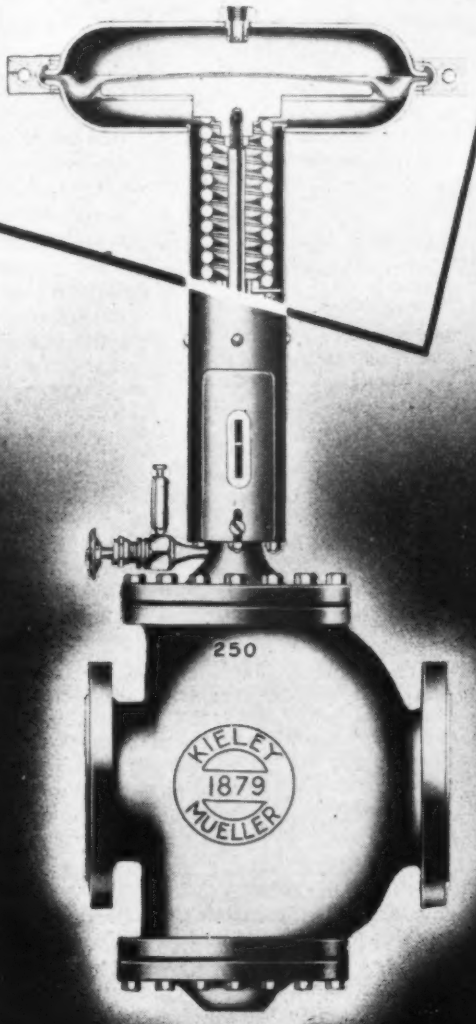

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Abstracts of Papers Presented Before

Division of Petroleum Chemistry, American Chemical Society

Atlantic City, New Jersey, April 14-18, 1947

Fundamental Studies on Separation of Hydrocarbons by Solvent Extraction. Henry J. Hibshman, Esso Laboratories, Standard Oil Development Company.

Thermodynamic Interrelation of Distillation, Vapor-Liquid Extraction and Liquid-Liquid Extraction. Equations relating the separation selectivities alpha, for distillation; beta, for liquid-liquid extraction; and gamma, for vapor-liquid extraction are derived thermodynamically. For separation of components which follow approximately the perfect solution and gas laws (such as hydrocarbons) the relationship alpha × beta = gamma is approximately true when the proper definitions of alpha, beta, and gamma are used.

Separation of Hydrocarbons According to Chemical Type and Molecular Weight by Solvent Extraction. Selectivities for separating hydrocarbons by vapor-liquid and liquid-liquid extraction processes were calculated from partial pressure data of pure hydrocarbons above a typical selective solvent, dimethyl phthalate. It was found that the boiling range of a mixture separable by a liquid-liquid process is roughly three times that separable by a vapor-liquid process and that the selectivities for separating hydrocarbons of equal boiling point according to chemical type (1) increase with boiling point, and (2) result in extraction in the following order: unsubstituted aromatics (benzene, naphthalene, etc.), alkyl-substituted aromatics, naphthenes, and paraffins.

Solid Solution Studies. Equilibria in the Binary System Cyclohexane-Methylcyclopentane. M. R. Cines, N. D. MacAllister, and L. R. Fruit, Phillips Petroleum Company, Phillips, Texas.

Continuing the calorimetric study of solid solutions between hydrocarbons, this paper presents the solid-liquid phase diagram for the binary system cyclohexane - methylcyclopentane. Methylcyclopentane exhibits limited solid-solubility in cyclohexane; it is miscible with the higher-temperature stable form, β, of cyclohexane and insoluble in the lower temperature form, α. The maximum concentration of the solid solution is 40 mol-percent methylcyclopentane. The eutectic mixture, 88 mol-percent methylcyclopentane-12 mol-percent α-cyclohexane, melts at —144.5° C.

With increasing concentration of solid solution, the temperature of the α-β transition of cyclohexane is lowered from —87.0° to —99.5° C. at the maximum concentration. Evidence for the existence of an invariant temperature, corresponding to a "peritectic," at —99.5 is presented.

The heat effects accompanying phase transformations have been evaluated wherever possible.

Evaluation of Viscosity-Temperature Characteristics of Hydrocarbons. R. T. Sanderson, Beacon Research Laboratory, The Texas Company.

The evaluation of kinematic viscosity-temperature characteristics of liquids is based on a viscosity-temperature number (V.T.N.). This is a number which designates the shape of the kinematic viscosity vs. temperature curve for all liquids which conform to the ASTM viscosity-temperature equation.

Simple relationships among viscosities, V.T.N.'s, the molecular weights of homologous hydrocarbons are presented. The effect of structure on viscosity-temperature of pure hydrocarbons is evaluated by V.T.N. Methods of applying the same basic principle to calculating viscosities of multicomponent mixtures and of oil-V.I. improver blends are described.

Analysis of Small Samples of Shale-Oil Naphtha by the Silica Gel Adsorption Method. G. U. Dinneen, C. W. Bailey, J. R. Smith, and John S. Ball, Petroleum and Oil-Shale Experiment Station, Laramie.

A method was developed for separating a 10-ml. sample of shale-oil naphtha into (1) paraffins and naphthenes, (2) olefins, (3) aromatics, sulfur compounds, and nitrogen compounds. The method is based on the selective adsorption of these groups of compounds by silica gel. Studies of adsorbabilities within these groups indicated that (1) relative adsorption of paraffins and naphthenes depends on concentration and molecular weight; (2) sulfur compounds are more strongly adsorbed than aromatics; (3) all nitrogen compounds used except pyrrole are more strongly adsorbed than the desorbent (2-propanol). With synthetic mixtures the method gave errors of less than 3 percent.

Adsorption. A new Tool in the Preparation of High-Purity Hydrocarbons. Alfred E. Hirschler and Senta Amon, Sun Oil Company.

Mixtures of isomeric saturated hydrocarbons, including geometric isomers, can be separated by adsorption on silica gel or activated carbon, and to a small extent on activated alumina. Data are given on the separation of many binary synthetic mixtures, including paraffin-paraffin, paraffin-naphthene, and naphthene-naphthene types. Most of these systems give S-type isotherms on silica gel and U-type isotherms on carbon. The effect of hydrocarbon type on adsorbability is discussed.

It is shown that many commercially obtainable and synthetic hydrocarbons may be obtained in high purity by adsorption. 2,3,3-trimethylpentane and 2,2,3-trimethylbutane were obtained in better purity than was previously possible by other methods. Its rather wide applicability, speed, and the use of very simple and inexpensive apparatus make adsorption one of the most powerful and convenient tools available for the separation and purification of saturated hydrocarbons.

It is pointed out that the results obtained can be reasonably explained in terms of the Freundlich adsorption isotherm.

Assembly and Testing of a 52-Foot Laboratory Adsorption Column. Separation of Hydrocarbons by Adsorption. Beveridge J. Mair, Andrew L. Baboriault, and Frederick D. Rossini, National Bureau of Standards. (API Research Project 6).

This report describes the assembly and testing of a large laboratory adsorption column, having a stainless-steel fractionating section ¾-inch (1.9 cm.) in inside diameter and 52.4 feet (16 meters) in length, and containing silica gel (largely 200- to 325-mesh in size) as the adsorbent. The procedure of operation, including regeneration of the adsorbent in place, is described. Results are given on the separation obtained with this column on (1) a synthetic mixture of n-dodecane, triethylbenzenes, and 1-methylnaphthalene, and (2) a sample of the kerosine fraction of petroleum.

Data are also given on the fractionation obtained in a glass adsorption column, 0.4 inch (1 cm.) in diameter and 26 feet (7.9 meters) in length, with the same silicia gel as the adsorbent, on binary mixtures of hydrocarbons selected to disclose differences in absorbability arising from differences in molecular size and structure.

Alkylbenzenes in the C_8 Fraction From Five Different Catalytic Petroleum Refining Processes. Anton J. Streiff and Frederick D. Rossini, National Bureau of Standards. (API Research Project 6).

This report gives the results of the analysis, by measurements of freezing points of appropriate mixtures, supplemented by analytical distillation, of the four individual C_8 alkylbenzenes (ethylbenzene, o-xylene, m-xylene, and p-xylene) occurring in the product from the following five different catalytic petroleum refining processes: (1) "hydroforming," (2) "two-pass fixed-bed," (3) "three-pass fixed-bed," (4) "low-temperature fluid," and (5) "high-temperature fluid."

Alkylbenzenes in the C_9 Fraction From Seven Representative Crudes. Alphonse F. Forziati and Frederick D. Rossini, National Bureau of Standards. (API Research Project 6).

This report presents the results of an

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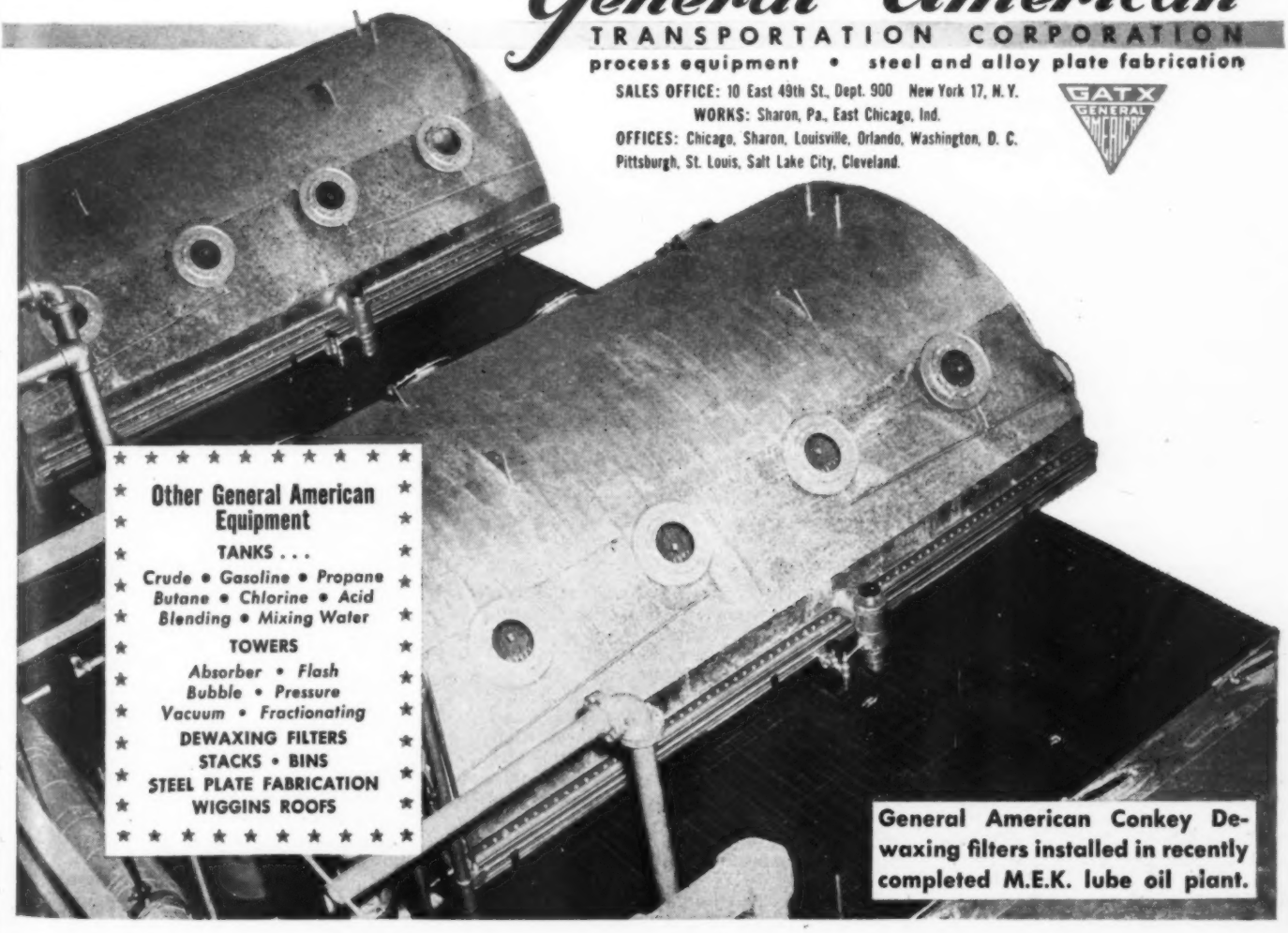

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analysis, by analytical distillation by the API Research Project 6, and by spectrographic examination by Socony-Vacuum Laboratories, Standard Oil Development Company, and Sun Oil Company, of the individual C_9 alkylbenzenes (except 1,2,3-trimethylbenzene) and of tert-butylbenzene occurring in the C_9 fraction from the following seven representative crudes (A) Ponca; (B) East Texas; (C) Bradford; (D) Greendale-Kawkawlin (E) Winkler, Texas; (F) Midway; (G) Conroe.

Hydrocarbons in the 102° to 108° C. Fraction of Petroleum. Augustus R. Glasgow, Jr., Charles B. Willingham, and Frederick D. Rossini, National Bureau of Standards (API Research Project 6).

This report describes the analysis of the hydrocarbons in the 102° to 108° C. aromatic-free fraction of petroleum, which is shown to be composed substantially of the following three compounds (normally boiling at the temperature indicated): ethylcyclopentane at 103.5° C.; 1,1,3-trimethylcyclopentane at 104.9° C.; 2,2-dimethylhexane at 106.8° C.

Separation and Determination of Aromatics From High-Boiling Petroleum Fractions by Adsorption. M. R. Lipkin, W. A. Hoffecker, C. C. Martin, and R. E. Ledley, Sun Oil Company.

This paper describes a method for the determination of volume-percent aromatics in petroleum fractions boiling above 200° C. (400° F.). The method has been tested on known blends of aromatic and saturated hydrocarbons separated from gas oil and lubricant fractions, and proved to be accurate within 1 percent. The sample is separated by silica gel into aromatic and nonaromatic fractions which may be used for further analysis. The method classifies aromatic olefins with the aromatics and classifies nonaromatic olefins with the paraffins and naphthenes. Analyses of lubricant fractions from several crudes are shown.

Determination of Total Aromatics in High-Boiling Petroleum Cuts by Ultraviolet Spectrophotometry. Alden P. Cleaves and Mildred E. Carver, Naca Aircraft Engine Research Laboratory.

A method is presented for determining the total aromaticity of petroleum cuts that boil in the range from 200° to 350° C., which extends higher than the range for which usual methods are suitable. This method is based on quantitative comparison of the ultraviolet absorption by the cut with that of a typical unadulterated sample of the aromatics extracted from the cut with silica gel, and isolated from the desorbents by distillation. The limitations on accuracy, arising largely in the extraction and concentration techniques designed to employ simple apparatus and to avoid a prolonged, tedious procedure, were determined by a series of tests on synthetic mixtures.

The method was applied to a 210° to 365° C. cut of a Hastings crude, and total aromatics were found to be 23 percent by volume. This value lies between the values for the same sample of 25.2 and 20.2 percent by volume determined by silica gel fractionation and by ASTM method ES-45a, according to data received from the Petroleum Experiment Station of the Bureau of Mines in Bartlesville.

Spectrophotometric Evaluation of Lubricating Oils. G. L. Clark and K. J. Pipenberg, Department of Chemistry, University of Illinois, and T. W. Culmer, Ohio Oil Company.

Continuing the study of the spectrophotometric evaluation of the stability and performance of oils, for which techniques and results have been published for quenching oils, successful application is made to a wide variety of lubricating oils. These include commercial oils heated under standard oxidizing conditions in the laboratory, oils from Underwood tests, and oils from actual engine service. Characteristic optical absorption curves are correlated with many standard chemical and physical tests—percent asphaltenes, percent carbon residue, percent chloroform-soluble and insoluble, percent petroleum naphtha-insoluble, copper-lead and cadmium-silver corrosion, neutralization number, viscosity. It is demonstrated that the spectrophotometric curves are a remarkably direct and simple method of evaluation and performance prediction, integrating in a single measurement the other methods.

The Mass Spectrometer in Organic Chemical Analysis. R. C. Taylor, R. A. Brown, W. S. Young, and C. E. Headington, The Atlantic Refining Company.

Data are presented to show that the mass spectrometer can be used to analyze products from the organic chemical laboratory as well as those from petroleum-refining and associated industries. Examples are given of the analysis of such mixtures as ethylene oxide, ethylene, and carbon dioxide; alkyl iodides; aliphatic chlorides, ethers, and hydrocarbons; silicanes; and water, ether, alcohol systems. The analysis of gas samples as small as 10^{-5} c. c. also are discussed and illustrated. The qualitative analysis of high-molecular-weight and organic compounds is mentioned, and some unique features of mass spectrometry emphasized.

Mass Spectrometer Analysis of Some Liquid Hydrocarbon Mixtures. R. A. Brown, R. C. Taylor, F. W. Melpolder, and W. S. Young, The Atlantic Refining Company.

Applications of the mass spectrometer method of analysis to normally-liquid hydrocarbons in the C_5 to C_8 range are discussed. Accuracies attainable for individual compounds in a number of different synthetic mixtures are given and typical analyses of several hydrocarbon fractions in gasoline boiling range are shown. It is concluded that a substantial number of paraffins, cycloparaffins, and aromatics occurring in this region may be individually determined. Olefins and cyclo-olefins, on the other hand, may be determined individually only to a limited extent at present.

Depolymerization of Butylene Polymers. F. G. Ciapetta, S. J. Macuga, and L. N. Leum, Atlantic Refining Company.

Catalytic depolymerization of C_8 homopolymers of isobutylene and its copolymers with n-butylene has been studied with the object of making pure isobutylene. Attapulgus clay was found to be an excellent catalyst for this depolymerization producing a C_4 stream from homopolymers of better than 99 percent isobutylene. With various condimers as feeds, isobutylene concentrations of 90 to 98 percent were obtained .

Mass spectrometer analyses of both gaseous and liquid products on two experiments showed that carbon skeleton isomerization of the C_8 olefins accompanied depolymerization. Evidence for the isomerization of 2,4,4-trimethyl-1 or 2-pentene into 2,3,4-trimethyl-1 or 2-pentene, 3,4,4-trimethyl-2-pentene into 2,3,3-trimethyl-1-pentene, and for changes of trimethylpentenes into dimethylhexenes was obtained. Explanations of depolymerizations and isomerizations by the carbonium-ion mechanism are given.

Selective Demythlation of Paraffin Hydrocarbons. Preparation of Triptane and Neopentane. Vladimir Haensel and V. N. Ipatieff, Universal Oil Products Company.

Branched-chain paraffin hydrocarbons react with hydrogen in the presence of a nickel or cobalt catalyst to give paraffins containing one less carbon atom and methane. The reaction involves the removal of specific methyl groups, since those attached to secondary carbon atoms are removed much more readily than those bound to the tertiary carbon atoms, while methyl groups attached to the quaternary carbon atoms are most stable. The reaction occurs within narrow ranges of experimental conditions.

Data are presented on the conversion of mixtures of 2,2,3-trimethylpentane and 2,3,4-trimethylpentane to triptane, 2,3-dimethylpentane, and 2,3-dimethylbutane. The results obtained on the demethylation of neohexane to neopentane also are given. The effect of operating variables such as pressure, temperature, and space velocity is discussed.

Production of 2,3-Dimethylbutane by Alkylation. R. B. Thompson and J. A. Chenicek, Universal Oil Products Company.

A process for the production of 2,3-dimethylbutane by the alkylation of isobutane with ethylene, using a liquid aluminum chloride-hydrocarbon complex as the catalyst and ethyl chloride as promoter, is described. The catalyst has a long life, is relatively inexpensive, and can be maintained at constant activity level such that high yields of 2,3-dimethylbutane are obtained.

Total alkylate yields of 270 to 300 weight-percent based upon ethylene consumed are obtained containing 68 to 75 volume-percent of 2,3-dimethylbutane. The catalyst life is approximately 85 gallons of total alkylate per pound of fresh aluminum chloride introduced from a saturator to the catalyst as a solution in the isobutane charge.

The following conditions are believed to be most desirable: a saturator temperature of 160° F. or lower in order to maintain catalyst activity at optimum value, reactor temperatures in the range 110° to 140° F., sufficient pressure for liquid-phase operation, 0.5 to 2.5 mol-percent ethyl chloride concentration, 15 to 25 minutes space time, and an isobutane-to-ethylene molar ratio of at least 4.5.

The composition of the catalyst affects the yield and quality of alkylate produced. A satisfactory alkylate may be obtained using catalyst with the following range of composition: 17 to 13 percent aluminum, 50 to 42 percent chloride, and 23 to 34 percent carbon.

Effect of Hydrogen on the Action of Aluminum Chloride on Alkanes. V. N. Ipatieff and Louis Schmerling, Universal Oil Products Company.

Molecular hydrogen at superatmospheric pressure has a surprisingly marked effect on the action of aluminum chloride on alkanes. When n-pentane is

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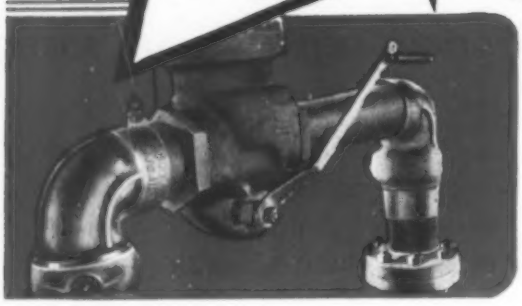

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heated with pure aluminum chloride at 125° C. in the presence of hydrogen, no reaction occurs; if nitrogen is used instead of hydrogen, autodestructive alkylation takes place, resulting in the formation of butanes, hexanes, and higher-boiling alkanes and in the conversion of the catalyst to a viscous red liquid. If, on the other hand, the reaction under hydrogen pressure is carried out in the presence of a promoter such as hydrogen chloride or water, isomerization to isopentane occurs in excellent yield with virtually no side reaction, and the catalyst remains crystalline.

Hydrogen does not prevent the autodestructive alkylation of heptane and higher-molecular-weight alkanes in the presence of pure aluminum chloride. However, if hydrogen chloride is added, destructive hydrogenation occurs and molecular hydrogen is consumed. This reaction has the advantage over autodestructive alkylation in that it results in a higher yield of isobutane and a smaller yield of catalyst complex (lower layer). Morever, the catalyst complex formed in the presence of hydrogen is more active than that formed in its absence indicating catalyst life.

Isomerization of Paraffins. Nature and Control of Side Reactions. B. L. Evering, E. L. d'Ouville, A. P. Lien, and R. C. Waugh, Research Laboratories, Standard Oil Company (Indiana).

In the isomerization of paraffins by means of $AlCl_3$ activated by HCl, side reactions occur which tend to deactivate the catalyst. Means are described by which these undesirable side reactions can be made practically negligible. These means include the use of gaseous hydrogen under pressure and the use of small amounts of aromatics or naphthenes. Quantitative data on the effect of these side-reaction inhibitors in the isomerization of pentanes and hexanes are presented. A new mechanism for isomerization, explaining the results, is proposed.

Catalytic Conversion of Xylenes to Toluene by Hydrogen Fluoride. P. L. Brandt, R. J. Lee, H. D. Radford, L. H. Klemm, and P. S. Drennan, Pan American Refining Corporation, Texas City, Texas.

The behavior of benzene-xylene mixtures and of xylene alone in the presence of hydrogen fluoride has been studied. Results obtained show substantial degrees of conversion of benzene-xylene mixtures to toluene under liquid-phase conditions at temperatures above about 100° C. and with quantities of hydrogen fluoride of the order of 200 weight-percent based on hydrocarbon. Simultaneously produced are more highly methylated benzenes. From xylene alone similar products plus some benzene were obtained. Data are presented showing the effect of ratio of reactants, quantity of catalyst, time, and temperature on the yield of toluene. Consideration of the experimental results indicates that benzene participates in the reaction.

Production of High-Cetane-Number Diesel Fuels by Hydrogenation. J. A. Tilton, W. M. Smith, and W. G. Hockberger, Esso Laboratories, Standard Oil Company of New Jersey, Louisiana Division.

Diesel fuels having cetane numbers above 70 and pour points below 30° F. have been produced experimentally by high-pressure destructive hydrogenation of paraffin wax, petrolatum, paraffinic

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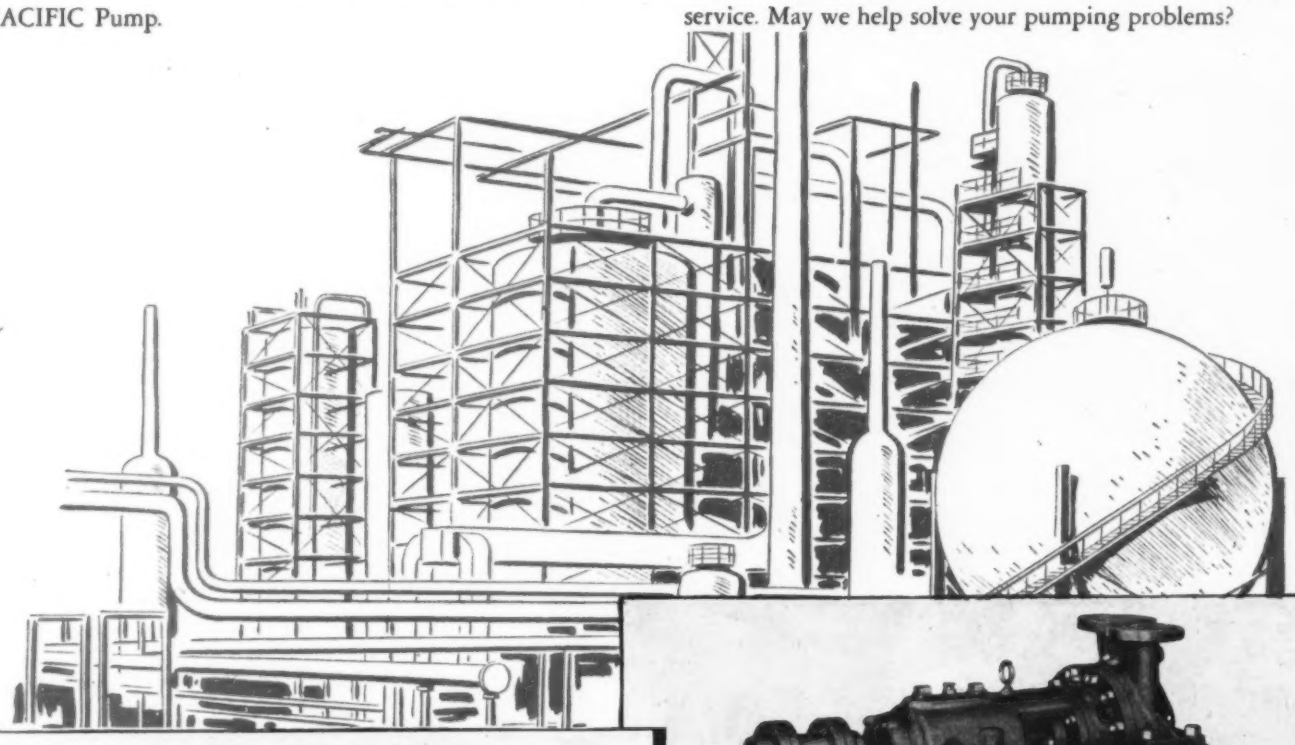

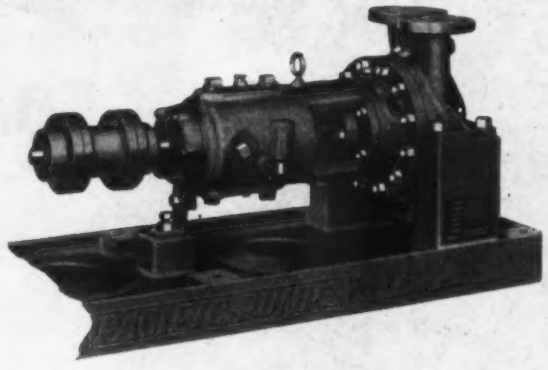

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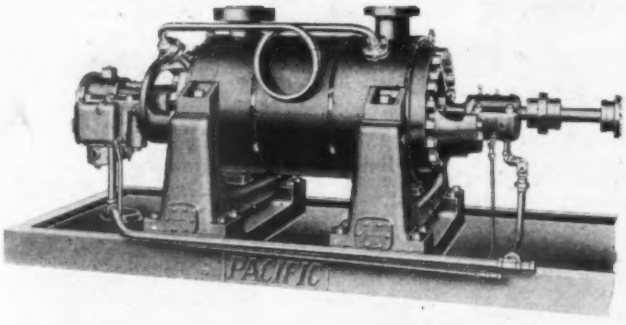

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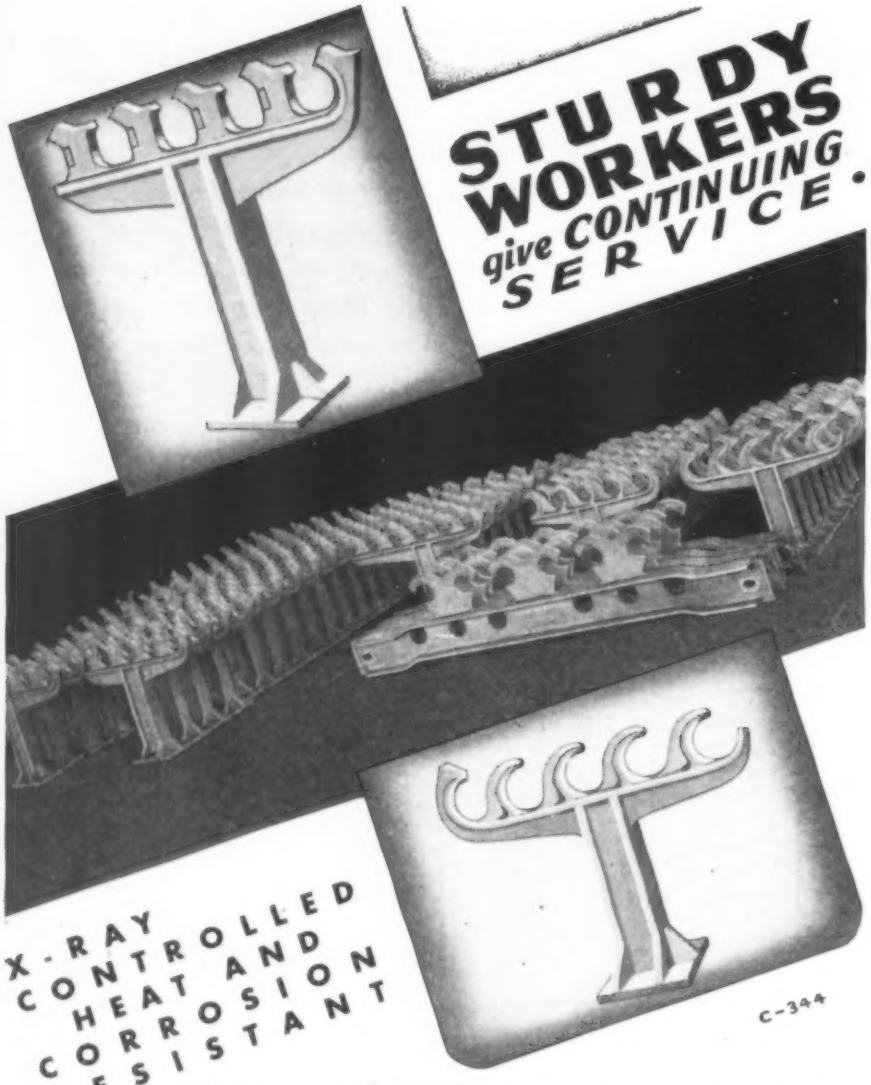

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gasoil, and by saturation hydrogenation of Fischer-Tropsch diesel fuel synthesized with an iron-type catalyst. These fuels contain chiefly straight-chain hydrocarbons having from 10 to 16 carbon atoms per molecule. Diesel fuels having cetane numbers of 50 to 55 have been produced by hydrogenation of virgin and catalytically cracked gasoil, both containing significant proportions of naphthene and aromatic hydrocarbons.

Production of Methylthiophenes From Pentanes and Sulfur. H. E. Rasmussen and P. P. Mellio, Socony-Vacuum Laboratories.

Reaction of butane with sulfur under specific conditions has been shown to produce thiophene. Under similar conditions iso- and n-pentane react to produce 3- and 2-methylthiophene, respectively. The process consists of preheating the pentane and the sulfur separately and reacting them at 900° to 1200° F. for a period ranging from a few seconds to a few tenths of a second depending on the temperature. The optimum yield is about 65 pounds of 3-methylthiophene from 100 pounds of isopentane and about 60 pounds of 2-methylthiophene from 100 pounds of n-pentane.

Adsorption-Desorption Isotherm Studies of Catalysts. III. Nitrogen and Stearic Acid Adsorption by Supported Catalysts and Their Components. Herman E. Ries, Jr., Marvin F. L. Johnson, and John S. Melik, Research and Development Department, Sinclair Refining Company, East Chicago.

Effects of supports on catalyst structure were studied with cobalt catalysts supported on diatomaceous earth, titania, and silica gel. Low-temperature nitrogen adsorption-desorption isotherms and stearic acid adsorption were determined. The data indicate that the unsupported material consists of finely-divided essentially nonporous particles and that a somewhat similar structure is obtained by supporting the catalyst on a nonporous titania. However a fine-pore catalyst structure of much greater area is obtained with a low-area diatomaceous earth support having large pores and an irregular structure. A high-area small-pore silica gel support also was studied.

Alumina and Alumina-Supported Catalysts. Electron Microscope Studies. P. G. Nahin and H. C. Huffman, Union Oil Company of California, Wilmington.

The following materials are examined with the electron microscope: fresh hydrous-gel Al_2O_3, gibbsite, baeyerite, boehmite, diaspore, corundum, β - "Al_2O_3," γ - Al_2O_3, θ - Al_2O_3, κ - Al_2O_3, a gel alumina catalyst support calcined at elevated temperatures, and fresh and thermally-deactivated molybdena-alumina and cobaltia-molybdena-alumina catalyst. Some qualitative correlations among the electron-optical, catalytic activities for hydroforming and hydrodesulfurization, x-ray diffraction, and surface-area data are treated in a preliminary fashion. It is shown possible to distinguish between the active and structurally deactivated states of the catalysts on the basis solely of particle size as revealed by electron micrographs.

Vanadium Oxide, a Hydrogenation Catalyst. V. I. Komarewsky, L. B. Bos, and J. R. Coley, Chicago.

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to the action of vanadium oxide-aluminum oxide catalyst at 400° and atmospheric pressure were converted to the corresponding paraffinic hydrocarbons with approximately quantitative yields. Diolefins were also readily hydrogenated to saturated hydrocarbons under the same reaction conditions. No olefins were detected in the reaction product.

Attempts to hydrogenate benzene with vanadium oxide have been unsuccessful to date at both atmospheric and high pressure (21 to 5 atmospheres) conditions.

The space velocity proved to be a critical variable for the hydrogenation reaction; a value of 0.050 liquid space velocity was found to be the maximum without serious loss of efficiency.

GAS AND FUEL CHEMISTRY

The following abstracts are of papers included in the program of the Division of Gas and Fuel Chemistry:

Thermal Efficiency of Coal Hydrogenation, Present and Future. L. C. Skinner, R. G. Dressler, C. C. Chaffee, S. G. Miller, and L. L. Hirst, U. S. Bureau of Mines, Coal Hydrogenation Demonstration Plant Division, Louisiana, Missouri.

The thermal efficiency of an improved coal hydrogenation plant is discussed in detail and the efficiencies are compared to a typical German hydrogenation plant. The new ideas and processes discussed are incorporated into a plant designed for improved thermal efficiency. The over-all heat efficiency of this plant is calculated to be 56.4 percent as compared to 28.9 percent for a typical German plant. The calculations are divided into 3 sections: hydrogenation proper, hydrogen manufacture, and power plant. Detailed data are presented to verify the results for each section. A comparison is made with the German efficiency for the individual sections with the following results:

	Improved Plant	German Plant
1. Hydrogenation proper	72.8%	51.1%
2. Hydrogen mfg.	58.5	27
3. Power plant	42.5	42
4. Over-all heat efficiency	56.4	28.9

The calculations are based on 100 tons per hour of moisture ash-free coal to hydrogenation proper with the following material quantities involved:

Total coal charged (as received)	227.3 T/hr.
Total coal charged (maf.)	194.7 T/hr.
Total coal to hydrogenation (maf.)	100 T/hr.
Total coal to H_2 production (maf.)	40.6 T/hr.
Total coal to power plant (maf.)	54.1 T/hr.
Gasoline produced	62 T/hr.
Liquefied fuel gases	13.3 T/hr.
Residuum fuel	11.0 T/hr.
Elec. energy req'd.	64,717 KWH
Steam req'd. (net)	502,150 #/hr.

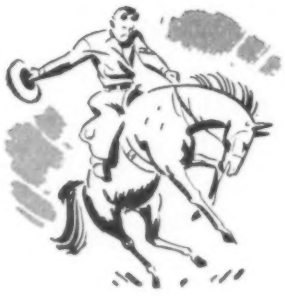
Flow sheets are included for each section showing complete heat and material balances.

The favorable increase in heat efficiencies shown, being accompanied by a considerable decrease in plant costs, indicates the necessity for pursuing an aggressive coal hydrogenation development study as a part of our national economic and security program.

Studies of the Fischer-Tropsch Synthesis. I. Tests of Cobalt Catalysts at Atmospheric Pressure. Robert B. Anderson, Abraham Krieg, Bernard Seligman, and Wm. E. O'Neill, U. S. Bureau of Mines, Pittsburgh.

The methods of preparation and testing of a precipitated cobalt-thoria-magnesia-kieselguhr (100:6:12:200) catalyst

BEAUMONT, TEXAS...



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is described. Usually tests with the same preparation of catalyst were quite reproducible, but tests with different catalysts prepared in the same manner showed very much wider deviations. Pelleted catalysts were shown to produce more light hydrocarbons and carbon dioxide than granular catalysts. Liquid hydrocarbons containing less than 6 percent dissolved wax comprised about 75 percent of the total hydrocarbons formed in these tests at atmospheric pressure. Experiments were made in which the flow of synthesis gas was varied with the temperature adjusted to give the same degree of conversion. Under these conditions the degree of unsaturation increased with increase in flow. Except for the change in unsaturation, the product distribution was not significantly different up to temperatures of 207° C. Overall activation energies of 24 to 26 kcal. per mol were found for the synthesis. The reaction mechanism and rate law of the synthesis is discussed briefly.

Effect of Various Catalysts on the Hydrogenolysis of Coal. M. Pelipetz, E. M. Kuhn, S. Friedman, and H. H. Storch, U. S. Bureau of Mines, Pittsburgh.

The liquid product resulting from the primary liquefaction of coal is of complex structure and consists of materials that are liquid at atmospheric conditions. Contained in this liquid product are dissolved and suspended semisolid and solid matter, the amount of which depends upon the degree of coal hydrogenation. The n-hexane-soluble fraction of the coal hydrogenation product is largely distillable and represents the most valuable product of coal hydrogenation. The fraction insoluble in n-hexane, but soluble in benzene, is undistillable and is of an asphaltic nature.

The experimental data presented show that asphalt is produced in the first step of coal cleavage, and with the progress of hydrogenolysis can be quantitatively reduced to form n-hexane-soluble material. Elimination of asphalt from the primary product of coal liquefaction was considered as the main object of this investigation.

It was expected that selected catalysts would be active in promoting hydrogenolysis of asphalts, the sluggish step in coal liquefaction. In the course of investigation it was found that Zn-Sb catalyst has the outstanding property of lowering the asphalt content, especially at low reaction temperature. Since tin proved to be one of the best catalysts in the primary cracking of coal, and Zn-Sb alloy showed activation in hydrogenolysis of asphalt, the idea of combined tin and Zn-Sb catalyst was conceived and tried.

The experimental results presented show that:

I. A combination tin and Zn-Sb catalyst enhances the hydrogenolysis of asphalt to a greater degree than Zn-Sb alloy alone and at the same time activates the primary cracking of coal to the same extent as tin alone.

II. Increasing the amount of the combined tin and Zn-Sb catalyst to 2 percent fails to improve materially the degree of coal liquefaction.

III. The presence of a vehicle or solvent in coal hydrogenation is undesirable in that larger amounts of asphalt appear in the product than when no vehicle is used.

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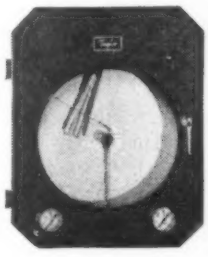

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under supervision of

DR. E. H. LESLIE and DR. H. B. COATS

The abstracts here presented are selected from the current literature of science and technology to afford reference to fundamental information not easily available to all readers. Abstracts of articles appearing in readily obtainable trade journals are not included.

Photostat copies of original articles will be supplied at cost by The Leslie Laboratories. Complete or limited bibliographies covering special topics by title, by abstracts, or in complete manuscript, will also be prepared and furnished at reasonable cost by the Laboratories.

Fundamental Physical and Chemical Data

Phase Equilibria in Hydrocarbon Systems. H. H. REAMER, K. J. KORPI, B. H. SAGE AND W. N. LACEY. *Ind. and Eng. Chem.*, **39** (1947) pp. 206-9.

The volumetric behavior of four experimental solutions of methane and n-butane, approximately evenly spaced as to mol fraction, was studied at seven temperatures from 100° to 460° F. and at pressures from 400 to 10,000 psia, in order to supplement previous measurements made on this system. Experimental data, interpolated to even values of pressure, are presented in tabular form as molal volumes and compressibility factors. Diagrams illustrating the behavior in some regions are included, and the new data are compared with those secured by another group of experimenters. A bibliography of 11 references is included.

Structural Determination of Paraffin Boiling Points. HARRY WIENER. *Jour. Am. Chem. Soc.*, **69** (1947) pp. 17-20.

The boiling points of organic compounds, as well as all their physical properties, depend functionally upon the number, kind and structural arrangement of the atoms in the molecule. Within any group of isomers, both the number and kind of atoms are constant, and variations in physical properties are caused by changes in structural interrelationships alone. A method for calculating the boiling points of the paraffin hydrocarbons is presented. This is applied to hydrocarbons from butanes to decanes and the results are given in tabular form. A complete set of the predicted boiling points for all of the nonanes is given in one table and this table and a second presents the boiling points for the decanes.

An Improved Flow Calorimeter. Experimental Vapor Heat Capacities and Heats of Vaporization of n-Heptane and 2,2,3-Trimethylbutane. GUY WADDINGTON, SAMUEL S. TODD AND HUGH M. HUFFMAN. *Jour. Am. Chem. Soc.*, **69** (1947) pp. 22-30.

U. S. Bureau of Mines has instituted, as part of its thermo-dynamic-research program, a project for the development and application of accurate methods for determining heat capacities of hydrocarbon vapors and their derivatives. The present paper describes the apparatus and experimental procedure now in use and presents molal heat capacities and heats of vaporization for n-heptane and 2,2,3-trimethylbutane. The apparatus used and the method of of measurement are described in detail. Data are presented in tabular and graphical form. It is believed that the measurements are accurate to within ±0.1 percent. For n-heptane the heats of vaporization reported at 90.48, 77.33 and 58.00° are 7715, 7938 and 8244 cal./mol respectively, and those for 2,2,3-trimethylbutane at 80.80 and 40.68°, 6918 and 7461 cal./mol.

The Vaporization of Fuels for Gas Turbines. Part I. The Bubble-Points and Dew-Points of Liquid Hydrocarbon Fuels. B. P. MULLINS. *Jour. Inst. of Petroleum*, **32** (1946) pp. 703-737.

The work on vaporization of gas-turbine fuels has been divided into two parts, first the bubble-points and dew-points of liquid-hydrocarbon fuels, and second, the heat quantities required to vaporize these fuels. The present paper deals with the first part of the problem. The search for new and improved gas-turbine combustion chambers naturally turns towards systems with perfect fuel atomization, an ideal that can be realized by pre-vaporization of the fuel. The author reviews the methods that have been used in the past 25 years for determining the bubble-points and dew-points of liquid hydrocarbons and applied these to standard fuels. Some practical determinations involving the preparation of "equilibrium solutions" were undertaken. Finally a theoretical method for calculating bubble-points and dew-points, depending on a knowledge of the true boiling-point distillation curve of the fuel in question was developed and applied to standard fuels.

The calculations cover a static pressure range of 10^{-2} to 10 atmospheres and have been extended to air-fuel ratios of 0:1 to 20:1. The results for an aviation gasoline, a kerosine, and a gasoil are given in the form of nomograms. A bibliography of 62 references is included.

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Molybdenum hexafluoride
Ruthenium tetroxide
Osmium octafluoride
Hexamethylacetone
5-Chloropentanone-2
m-Ethyltoluene
Diphenyl hydroxylamine
Dimenthylhydroxylamine
1,2,3,4-Tetrachlorobenzene
Methylisopropylcyclopentanes
Methylethylcyclohexanes
1-Butyne
1-Penten-4-yne
2,3,5,6-Tetramethylbenzaldehyde

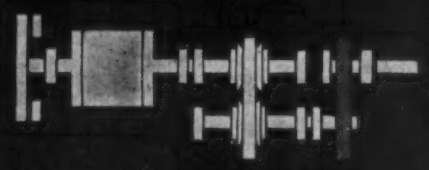
Chemical Compositions and Reactions

The Nitrogen Compounds in Petroleum Distillates. XXVI. Confirmation of Presence of Bx-tetrahydroquinolines in California Petroleum. P. L. PICKARD AND H. L. LOCHTE. *Jour. Amer. Chem. Soc.*, **69** (1947) pp. 14-6.

In continuing the study of the basic nitrogen compounds present in California petroleum, the 230-232° C.-boiling-range material was investigated. The methods used in the work are described. A $C_{11}H_{15}N$ base was isolated. Ultraviolet absorption studies indicate that the natural $C_{11}H_{15}N$ base is an alkylated Bx-tetrahydroquinoline, thus confirming the presence of this type of base in petroleum. 3-Cyclopentyl-5-methylpyridine were synthesized and shown to be different from the $C_{11}H_{15}N$ base isolated from petroleum. 3-Ethyl-5-methylpyridine were synthesized and shown to be identical with the $C_8H_{11}N$ base isolated from petroleum.

Decenes Formed from t-Amyl Alcohol and from 2-Methyl-2-butene. Composition of the Hydrogenated Products. GEORGE C. JOHNSON. *Jour. Amer. Chem. Soc.*, **69** (1947) pp. 146-9.

The author notes that although the formation of decenes from t-amyl, i-amyl and optically-active amyl alcohol and also by the dimerization of isopentenes in the presence of catalysts has been studied for a century, there still exists doubt as to the structures of the resulting decenes. The work of which the present paper is a report was undertaken to establish decene structures more definitely. In these studies decenes were formed from t-amyl alcohol in the presence of sulfuric acid and from 2-methyl-



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2-butene in the presence of an alumnia-silica catalyst. The procedure for investigating the structures comprised the hydrogenation of the decenes to decanes and the subsequent analysis of the decanes, by efficient fractional distillation accompanied by determination of the physical properties of the fractions. It was concluded that the decanes formed by hydrogenating the decenes made from t-amyl alcohol in the presence of sulfuric acid below 75° are 2,2,3,4-tetramethylhexane and 3,3,5-trimethylheptane together 31.1 percent by volume; 2,3,4,4-tetramethylhexane 36.9 percent; 2,3,3,4-tetramethylhexane 30.3 percent; 2,3,4-trimethyl-3-ethylpentane 1.7 percent. The decanes formed by hydrogenating the dimer of 2-methyl-2-butenes formed in the presence of an alumina-silica catalyst at 110° C. are mostly 3,3,5trimethylheptane with lesser amounts of 2,3,4,4-tetramethylhexane and 2,3,3,4-tetramethylhexane. Physical properties of five decanes and one dodecane are reported.

Monoalkylbenzenes by Vapor-Phase Alkylation with Silica-Alumina Catalyst. A. A. O'Kelly, J. Kellett and J. Plucker. *Ind. and Eng. Chem.*, **39** (1947) pp. 154-8.

Studies were made of the alkylation of benzene with olefins, such as ethylene, propylene, and propylene-propane mixtures for the formation of ethylbenzene or of cumene. The alkylation was conducted in the vapor phase over a silica-alumina catalyst by both batch and continuous methods. Catalysts were of the type used in commercial catalytic cracking. The utilization of ethylene was found to be favored by increased reaction time, increased temperature, and increased molar ratio of benezene to ethylene. Small amounts of polyethylbenzenes produced can be recycled in the charge stream to give increased yields of monoethylbenzene based on ethylene and benzene consumed. The catalyst indicates long life and sustained activity under the conditions used. A cyclic operation such as is used in catalytic cracking is adaptable to the production of monoethylbenzene in which air regeneration of the catalyst is carried out at temperatures in the same range as the reaction temperatures. A flow diagram is presented of the continuous low-pressure alkylation unit. Experimental data are presented in detail in tabular form and a bibliography of 17 references is included.

The Synthesis and Physical Properties of Tetraethylmethane. A. Wesley Horton. *Jour. Am. Chem. Soc.*, **69** (1947) pp. 182-3.

A three-step synthesis of the nonane-tetraethylmethane is given and the principal properties determined experimentally on a carefully purified sample are reported. Empirical equations are given relating molecular structure to boiling point, density, and refractive index for the symmetrical series $(CH_3)_{4-n}C(C_2H_5)_n$ are presented.

Manufacture: Processes and Plant

Batch-Distillation Nomograph. Melvin Nord. *Ind. and Eng. Chem.*, **39** (1947) p. 232.

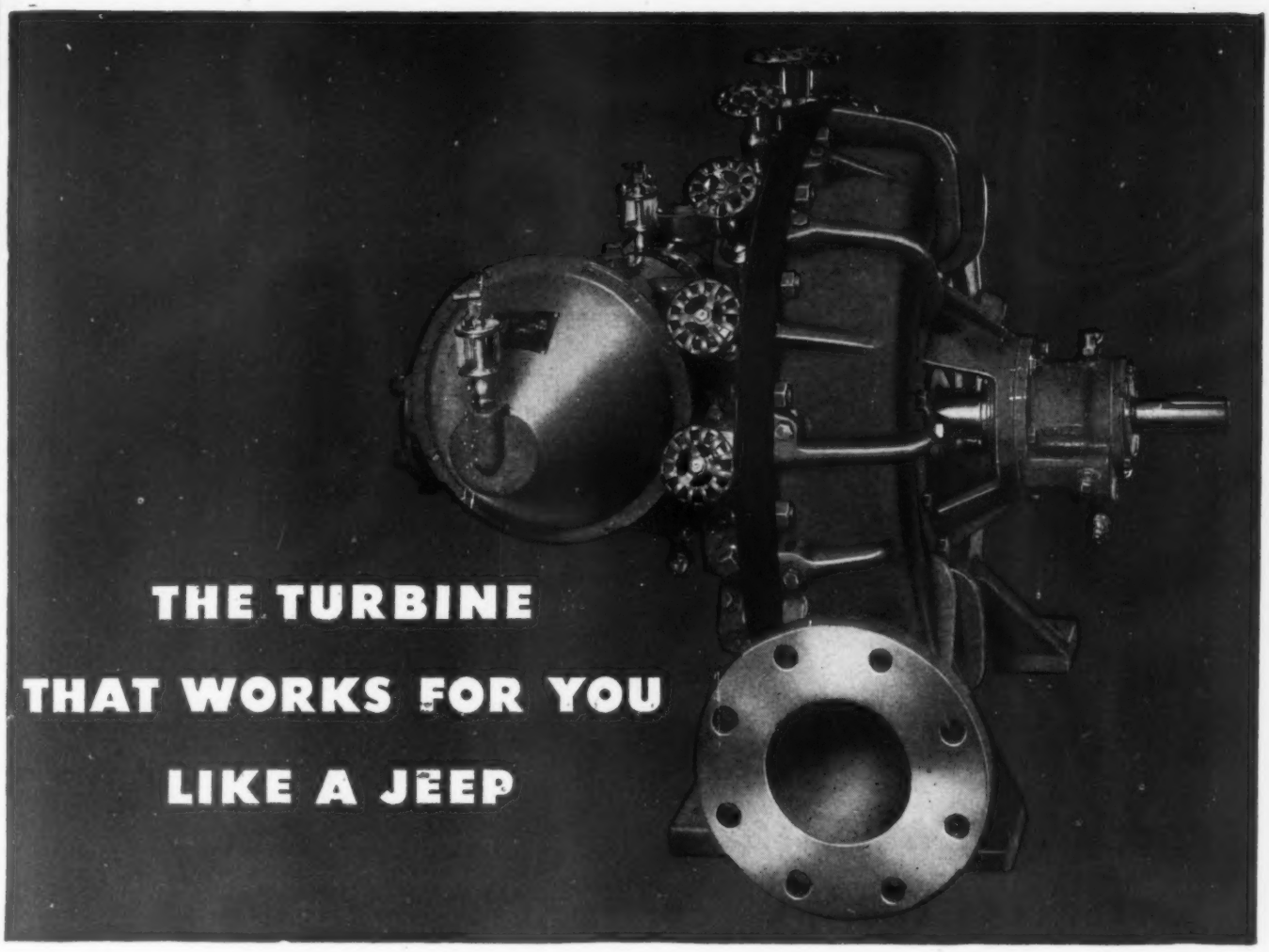
In the theory of simple batch distillation of binary solutions Rayleigh's equation applies. In this x_1 and x_2 are the mol fractions (or weight fractions) of the light component in the original charge and the residue. In order to solve the equation for x_1 or x_2, a trial-and-error solution is necessary. It is possible, however, to construct a nomograph that will solve these equations without trial and error. The author presents such a nomograph and illustrates its application to a particular problem. If more precise results are desired than can be obtained with a nomograph of moderate size, the values obtained by use of the nomograph can be used as those of the first approximation in a trial-and-error solution.

Naphthas from Fluid Catalyst Cracking. C. E. Starr, Jr., J. A. Tilton and W. G. Hockberger. *Ind. and Eng. Chem.*, **39** (1947) pp. 195-202.

The compositions of naphthas derived from cracking with fluid catalyst vary within wide limits depending upon changes in the operating conditions, types of catalyst employed, and the feed stocks used. In the present article the compositions of nine naphthas are given to illustrate the types of products obtained at low and high cracking temperatures, employing clay and synthetic catalysts with several paraffinic and naphthenic feed stocks. Naphthas from naphthenic feed stocks are characterized by high yields on feed together with high aromatics concentrations; yields of toluene and C_8 aromatics are higher than are obtained by cracking paraffinic-type feed stocks under comparable conditions. Yields of naphthenes and cyclo-olefins tend to be higher in naphthas from naphthenic feed stocks, but yields of C_4-C_7 paraffins and olefins are reduced. The data show that a number of valuable hydrocarbons, such as toluene, are present in the cracked naphthas to an extent that makes recovery feasible. Using fluid-catalyst cracking for the production of high-quality fuels also permits the concurrent production of a number of hydrocarbons that are individually valuable as raw materials for chemical manufacture, as for example, isobutane, isobutylene, normal butenes, which are employed in alkylation processes for the production of aviation fuels of high octane number or in chemical processes for the manufacture of synthetic rubber. The data secured in the work are presented in 9 tables and a bibliography of 13 references is included.

Optimum Dilution in Viscous Liquid Filtration. E. J. Reeves. *Ind. and Eng. Chem.*, **39** (1947) pp. 203-6.

The viscosity of a liquid subjected to filtering operations is one of the major factors governing filtering rates. If all other variables are constant, the rate of filtration is inversely proportional to the absolute viscosity of the filtrate. The problem resolves into how to increase the amount of solvent-free material in the filtrate. The optimum dilution is determined normally on the basis of plant experience; however, it can be calculated mathematically from the known properties of the involved materials. A useful relationship is derived for calculating optimum dilution in viscous liquid filtration. This relationship is based on an empirical equation that was developed for expressing the viscosity of the filtrate as a function of the solvent dilution. By use of the mathematical equation the method can be adapted to dilution filtration of any viscous liquid. Observed and calculated data are in good agreement. The feature is illustrated by



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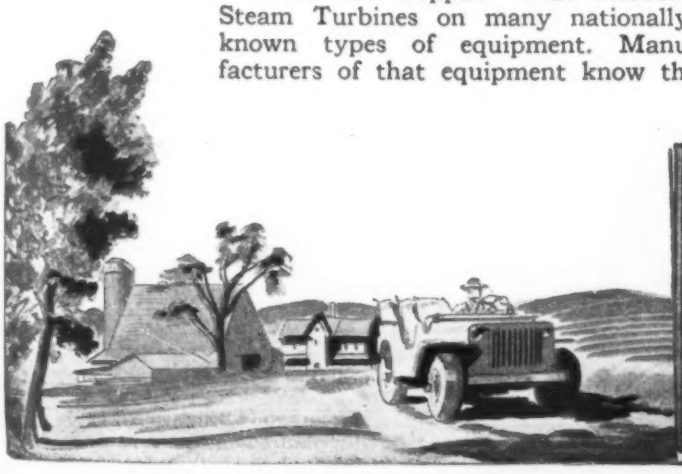

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application to neutral oil and Mid-Continent residuals. The data are presented in detail in five tables and five figures.

Multistage Separation Processes. MAXSON BENEDICT. *Trans. Am. Inst. Chem. Engrs.*, **43** (1947) pp. 41-60.

The application of multi-stage separation processes in engineering work has increased greatly in recent years. Examples of important new applications are the use of extractive distillation in butadiene and toluene production and the use of thermal diffusion, gaseous diffusion and the gas centrifuge in the concentration of Uranium 235. The author reviews and describes briefly these new types of processes, giving examples of their application, and compares them with ordinary distillation, and other familiar separation processes. The theory and basic constants of each type of process are discussed, such as the characteristic separation factor and energy consumption, and the advantages and disadvantages of each process are considered. A bibliography of 22 references is included.

Production of Butadiene from Alcohol. W. J. TOUSSAINT, J. T. DUNN AND D. R. JACKSON. *Ind. and Eng. Chem.*, **39** (1947) pp. 120-5.

During the war emergency a substantial part of the butadiene for use in making synthetic rubber was produced from ethanol. Since ethanol can be made from ethylene, a by-product of petroleum-cracking operations, this process is of direct interest to the petroleum industry. Development of the alcohol process was facilitated by the recognition that crotonaldehyde is the intermediate in the Ostromislensky reaction, in which alcohol and acetaldehyde are converted to butadiene. Initially a process was developed for converting crotonaldehyde and ethanol to butadiene using silica gel as a catalyst, but suitable modification of the catalyst afforded equally efficient production of butadiene from acetaldehyde and ethanol. Furthermore, by incorporating dehydrogenating components in the catalyst, the Lebedev type of process was effected by converting ethanol more directly to butadiene. The preferred catalyst for the acetaldehyde-ethanol reaction was silica gel impregnated with tantalum oxide. With this catalyst efficiencies of about 67 percent yields from ethanol were obtained in the laboratory and the results were substantially reproduced in plant operation. Zirconia supported on silica gel was considered as a possible replacement catalyst, because zirconia was more readily available and the yield of butadiene was only somewhat less than that obtained using the tantalum catalyst. The by-products from the process comprise hydrocarbons, aldehydes, ketones, esters, ethers, alcohols, and some other materials. The more important of these—namely, ethylene, butenes, ethyl ether, and butanol—were recovered on a plant scale in quality suitable for utilization.

The ethanol-acetaldehyde process fulfilled its mission in providing a large supply of butadiene in the shortest possible time from what was a surplus material at that time.

Selecting Chemical Pumps. C. A. RICHARDSON. *Chem. Eng. Progress*, **1** (1947) pp. 17-20.

The same amount of thought and engineering skill should be used in the selection of a pump as in the selection of any other engineering equipment item. The

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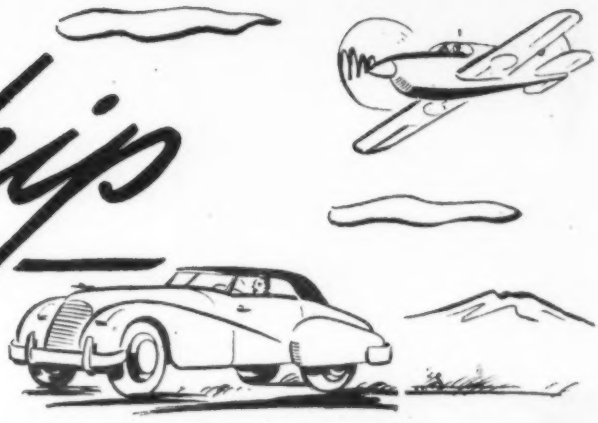

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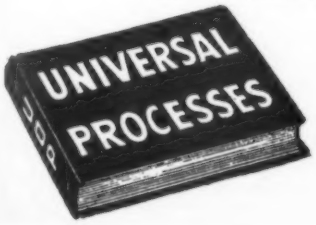

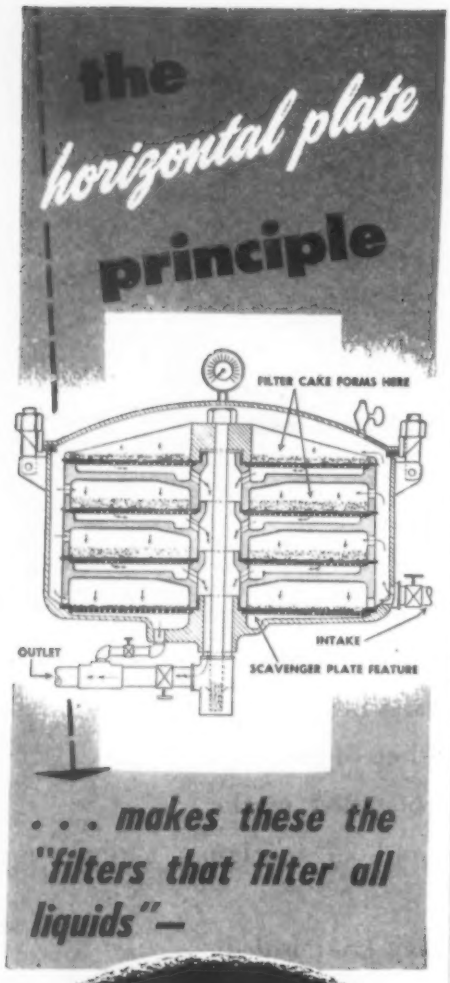
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purpose of the article is to indicate some of the more important construction details and operating factors that should be considered in the selection of pumps, particularly where both corrosive and abrasive liquids are to be handled. Advantages of the double-suction pump with horizontally split casing over the single-suction vertically split casing pump are given. The main disadvantage of the horizontally-split-case pump is high initial cost and high replacement cost for repair parts. The question of the selection of closed versus open impellers in centrifugal pumps is considered. In general, closed impeller pumps should be used unless the liquid pumped deposits scale on the impeller surfaces or contains solids that will plug a closed impeller. Wearing rings, shafts, and stuffing boxes are discussed.

Proportioning Pumps — Their Construction, Performance, and Application. ROLLAND MCFARLAND, JR. *Chem. Eng. Progress,* **1** (1947) pp. 12-16.

The words "proportioning" and "metering" are defined and distinguished as applied to adjustable-stroke, positive-displacement pumps. A proportioning pump is a metering pump used to proportion one liquid to another. The design requirements of metering pumps are outlined, because of the close-clearance design and accurate check-valve action these pumps have high volumetric efficiency. Factors that affect metering-pump performance are reviewed, including viscosity, suspended solids, vapor pressure, and pumping temperature. Attention to proper maintenance is urged, and two examples are given of poor pump performance caused by lack of proper maintenance.

The Design of Jet Pumps. A. EDGAR KROLL. *Chem. Eng. Progress,* **1** (1947) pp. 18-24.

In spite of the wide application of jet pumps, or ejectors, as they are sometimes called, few design data are available in the literature. The purpose of the paper is to provide the engineer with a simple method for the design of single-stage steam- and air-operated jet pumps, having maximum efficiency for handling gases. The discussion covers diffuser entrance, diffuser throat, distance of nozzle outlet to diffuser throat, induced fluid entrance, diffuser outlet, tail piece, and nozzle. The design procedure is applied to an example.

Products: Properties and Utilization

Review of the Chemical Structure of Fuels and Their Detonation in Engines. A. G. MAZURKIEWICZ AND V. G. OBERHOLZER. *Jour. Inst. of Petroleum,* **32** (1946) pp. 685-702.

The author notes the large accumulation of data published by many laboratories working on the subject of combustion during the war and considers that a need has arisen for a general review of the results. The first part of the article is devoted to a discussion of various engine-testing methods. Following this the knock characteristics of fuels or hydrocarbons of different types is considered. The effect of chemical structure is discussed and data on the various hydrocarbons are presented in tabular and graphical form. The aromatic amines, which were widely used during the war, are discussed particularly with their effect on knocking in rich mixtures. The final part of the article is devoted to a discussion of the results reviewed and presented. Those hydrocarbons which on oxidation yield ketones rather than aldehydes are best from an anti-knock standpoint. A bibliography of 32 references is included.

Resinous Products from Petroleum Polymer Sulfurization. M. GLENN MAYBERRY, PAUL V. MCKINNEY AND HARRY E. WESTLAKE, JR. *Ind. and Eng. Chem.,* **39** (1947) pp. 166-7.

Polymers from clay refining towers used in removing diolefins from cracked gasoline were studied. Resins ranging in properties from soft to brittle solids were formed when petroleum tower resins were heated with sulphur for various lengths of time and at various temperatures. The properties of typical products so made are given. The sulphurized products are resistant to salts and low concentrations of sulphuric acid. Nitric acid attacks them to form hard, brittle, and easily crumbled materials. Neither sodium hydroxide nor hydrochloric acid has any apparent effect, but ammonia and calcium hydroxide have slight bleaching action. All of the products are too brittle to be of use by themselves, but they can be compounded to give a mastic composition that will flow to fill a simple mold cavity completely if heated under pressure. Fillers, such as whiting, asbestos, wood flour, carbon black, and the like, were found to be satisfactory for the preparation of mastics suitable for floor tile, for example. Pigments can be added to give dark shades of red, brown, green, and black. The dark color of the resin itself prohibits lighter colors. Various methods of modifying the mastic to obtain an inexpensive molding composition were inconclusive. Although the products have low tensile and impact strengths, addition of cyclopentadiene to the original reaction mixture gave a product with both properties improved.

Performance of Butyl Inner Tubes. I. E. LIGHTBOWN, L. S. VERDE AND J. R. BROWN, JR. *Ind. and Eng. Chem.,* **39** (1947) pp. 141-6.

In severe service tests Butyl-rubber tubes have been shown to hold air 8 to 10 times better than natural-rubber tubes. Test results demonstrate that the maintenance of proper inflation pressure, afforded by Butyl tubes because of their superior air retention, results in an increase in tire-tread life of 10 to 18 percent, depending on the test conditions. Butyl tubes can be made that show less "growth" than natural-rubber tubes. Butyl inner tubes age well in service, maintaining their original physical properties better than natural-rubber tubes. This superior aging results in an inner tube with better puncture and blowout, or tear, resistance, which, in turn, provides a greater degree of safety for the motorist. The data secured in the course of the work are presented in considerable detail in tabular and graphical form and a bibliography of 12 references is included.

Constant Ratio Still Head. L. E. LLOYD AND H. G. HORNBACHER. *Anal. Chem.* **19** (1947) pp. 120-3.

The authors review and discuss the subject of laboratory still heads and give

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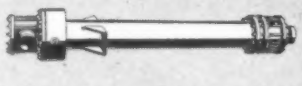

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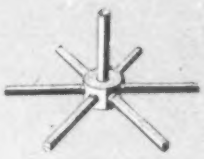

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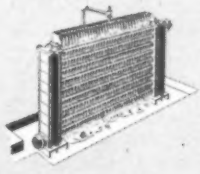

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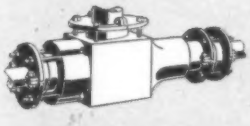

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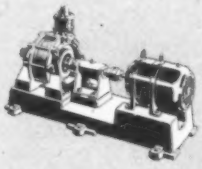

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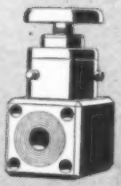

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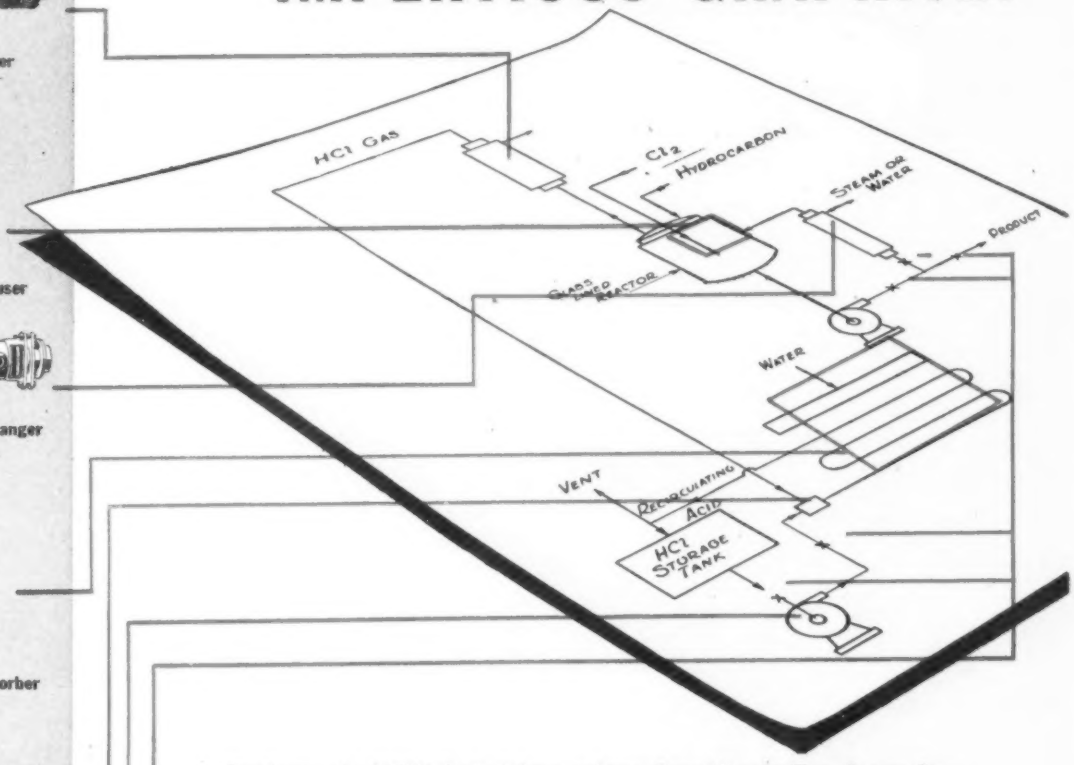

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a bibliography of 46 references on the subject. They note that several constant-reflux-ratio stillheads have been devised that have given improved column efficiency and more reproducible distillations with less attention from the operator than when using former types of heads. These heads are based on the principle of using two condensing surfaces in parallel—one to furnish reflux, the other to give product.

Microdetermination of Tetraethyl Lead in Gasoline. B. E. GORDON AND R. A. BURDETT. *Anal. Chem.*, **19** (1947) pp. 137-40.

A micromethod for the determination of tetraethyl lead in gasolines is based upon decomposition with iodine, followed by the volumetric determination of precipitated lead chromate. Elapsed time required is considerably shorter than that of conventional methods, and the volume of the sample needed is only 1 to 5 ml. The accuracy and reproducibility of the method conform to ASTM specifications and requirements.

Capillary-Type Viscometer. D. P. SHOEMAKER, EARL HOERGER, R. M. NOYES AND R. H. BLAKER. *Anal. Chem.*, **19** (1947) pp. 131-2.

A capillary-type viscometer for taking measurements of solutions containing volatile solvents was designed and tested. Because the solution does not need to be transferred from the container in which it is prepared, measurements made with this viscometer on concentrated solutions of large molecules are more reproducible than those made with an Ostwald-type capillary viscometer. Because of the small volume of the new viscometer, viscosity determinations can be made by using smaller samples than those required for the falling-ball viscometer. The simplicity of the device and its freedom from errors caused by concentration changes recommend its application to the study of a variety of substances in solutions containing volatile solvents.

Analysis of Naphthalene-Tetralin-Decalin Mixtures. W. J. CERVENY, J. A. HINCKLEY, JR., AND B. B. CORSON. *Anal. Chem.*, **19** (1947) pp. 82-86.

A rapid and simple method for the analysis of naphthalene-tetralin-decalin solutions is described and gives determinations within ±1 percent for each of the three components. Two measurements are required: first, the heat of reaction of the sample with nitrating acid, and second, the temperature at which naphthalene crystallizes upon cooling the sample. The method is applicable to all solutions of naphthalene, tetralin, and decalin. An alternate method that has certain advantages of simplicity of principle and operation, comprises separating the sample by distillation into an overhead and a bottom and then measuring the specific dispersions on each of these fractions.

Determination of Mono- and Dialkylacetylenes. CHARLES D. WAGNER, THEODORA GOLDSTEIN AND EDWARD D. PETERS. *Anal. Chem.*, **19** (1947) pp. 103-5.

A method is described for the determination of four and five carbon atom, mono-and dialkylacetylenes, in the presence of paraffins, olefins, or diolefins. The method is based on the reaction of the acetylenes with methanol in the presence of a catalytic mercuric oxide-boron trifluoride combination to produce ketals which are subsequently hydrolyzed to ketones. Allenes and high concentrations of peroxides and cyclopentadiene interfere, but they are the only materials known to cause interference. Interference by carbonyl compounds is obviated by suitable blank determinations, omitting the catalyst. Although the procedure does not ordinarily give stoichiometric results, recoveries of approximately 92 percent are consistently obtained, and therefore it is possible to use an empirical correction factor of 1.09. Analyzing samples containing as little as 0.5 percent acetylenes, the accuracy is ±3 percent and on samples containing 0.1 percent acetylenes, the accuracy is ±10 percent.

Microchemistry. ROWENA PYKE, ALLAN KAHN, AND D. J. LEROY. *Anal. Chem.* **19** (1947) pp. 65-66.

Although it is relatively easy to separate a gas sample into fractions having the same carbon number, it is much more difficult to make a precise analysis of these fractions in terms of olefin content. Two new procedures are described, using the Blacet-Leighton gas-analysis microapparatus. In one method, the absorbent was a paste made from 3 c. c. of powdered mercuric acetate and 2 c. c. of a 1-percent solution of boron tribluoride ethyl etherate in ethylene glycol. In the second method the absorbent was made from 3 c. c. of powdered mercuric acetate, 1.5 c. c. of water, and approximately 1 gram of mercuric nitrate. Little difference was found between the two methods in the case of ethylene-ethane mixtures, but the greater versatility of the hydroxy mercurial method recommends its use in most instances.

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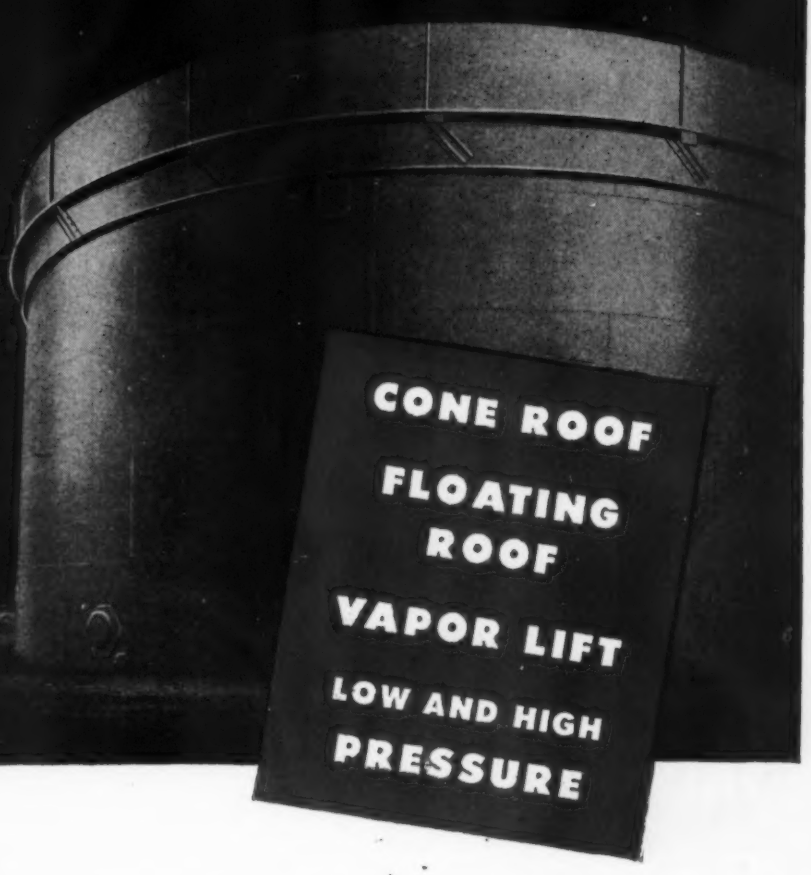

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U.S.P. 2,414,252. Solvent Separation of Hydrocarbons. H. V. Ashburn to The Texas Company.

A hydrocarbon mixture containing saturated and unsaturated hydrocarbons is extracted with a normally solid polyolefin glycol, such as Carbowax, if desired in the presence of water, at a temperature and pressure sufficient to maintain the solvent and hydrocarbons in the liquid phase. The solvent phase comprises unsaturated hydrocarbons dissolved in the bulk of the solvent. The other phase comprises saturated hydrocarbons mixed with a small amount of the solvent. The phases are separated and the hydrocarbons are recovered from the solvent. The process can also be used for separating aromatic hydrocarbons from aliphatic and/or alicyclic hydrocarbons.

U.S.P. 2,414,259. Treatment of Hydrocarbon Materials. M. M. Holm to California Research Corporation.

Hydrocarbon material containing a relatively minor quantity of combined F, such as the material resulting from treatment of hydrocarbon with an F-containcatalyst, is subjected to the action of CaO or MgO, or both under conditions effective to remove the organic fluorine-containing compounds, such as a temperature of 100-700° F.

U.S.P. 2,414,371. Controls for Isomerization Systems. N. Fragen and C. W. Nysewander to Standard Oil Company (Indiana).

A hydrocarbon stream of substantially constant composition containing dissolved HCl, ethane, gases lower boiling and gases higher boiling than ethane, is continuously introduced at substantially constant rate into a separation zone wherein a portion of the dissolved HCl is separated as HCl vapor or gas along with vapors or gases of the said hydrocarbons. A liquid stream of constant composition is continuously withdrawn from this separation zone and introduced into a stripping zone. At least part of this liquid stream is introduced near the top of the stripping zone where a lower temperature is maintained than at the base of this zone. The heat supplied to the stripping zone is sufficient to vaporize dissolved HCl and hydrocarbon gases of the type described. The temperature at the top of the stripping zone is sufficiently low to condense most but not all of the hydrocarbons which are boiling higher than ethane. HCl gas along with a substantial proportion of the lower-boiling hydrocarbons and a minor proportion of the higher-boiling ones is withdrawn from the top of the stripping column at a constant volume rate by regulating the amount of heat introduced into this zone. The withdrawn gaseous or vaporous stream is contacted with a stream of low-boiling paraffin hydrocarbons under such conditions that a major part of the HCl gas and higher-boiling hydrocarbons is absorbed while unabsorbed gases or vapors are vented at the top of the absorption zone. A liquid hydrocarbon stream containing ethane and HCl is continuously removed from the base of the stripping zone. The amount of ethane removed in this way is substantially in excess of that vented at the top of the absorption zone. The withdrawn liquid is freed from HCl. The process is employed in connection with processes for isomerizing hydrocarbons by means of $AlCl_3$-hydrocarbon complex catalysts promoted by HCl.

U.S.P. 2,414,402. Removal of Foam-Producing Substances from Furfural Employed in Extractive Distillation of Hydrocarbons. G. Thodos and C. F. Weinaug to Phillips Petroleum Company.

Furfural used in hydrocarbon selective-solvent extraction processes, particularly in processes for recovering n-butene and/or butadiene from more saturated C_4 hydrocarbons by extractive distillation, contains foam-producing impurities acquired from the low-boiling aliphatic hydrocarbons treated. To remove these impurities the furfural is subjected to liquid-liquid extraction with an aliphatic hydrocarbon immiscible with furfural but able to dissolve said foam-producing impurities. The two phases formed are separated.

U.S.P. 2,414,586. Distillation of Hydrocarbonaceous Solids. G. Egloff to Universal Oil Products Company.

Hydrocarbonaceous solid material, such as coal or oil shale, is introduced in finely divided state into a confined distilling zone, where a bed of this material is maintained in turbulent fluid state. A stream of non-oxidizing fluid preheated to a temperature adequate for the desired distillation is introduced upwardly into said bed to effect distillation of volatiles from the solid material. The resulting fluid is removed from the upper portion of said bed. Heavier solid particles are separated from the withdrawn fluid stream and returned to the bed. Regulated quantities of the solid particles are removed from the lower region of the bed and commingled with the now oxidizing fluid. The mixture obtained is again preheated and passed back to the distilling zone. Valuable volatile constituents are recovered from

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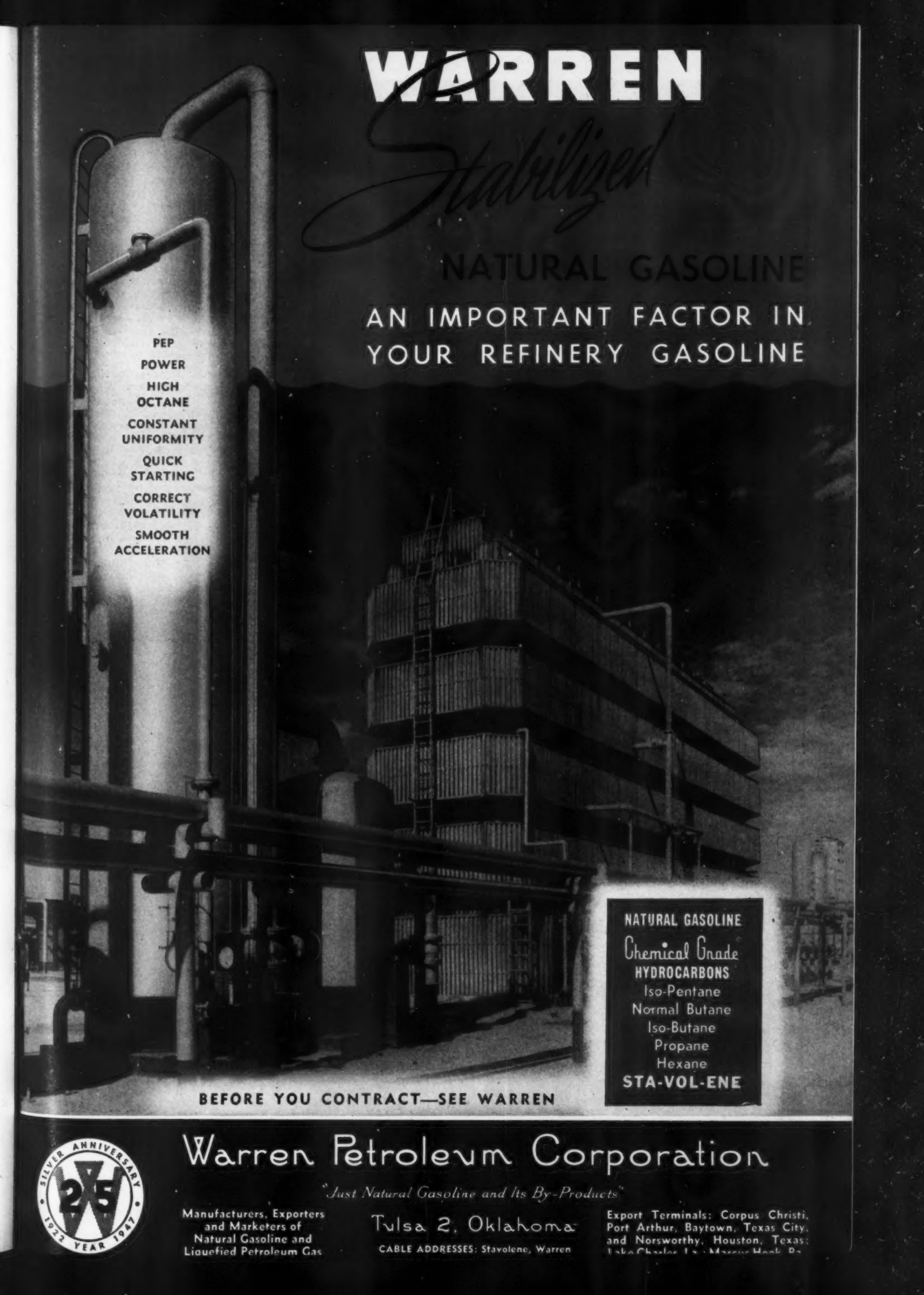
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the hydrocarbonaceous solids. Gaseous or liquid fractions separated from the recovered products of the distilling process may be employed as the non-oxidizing fluid.

U.S.P. 2,414,639. Process for Concentrating Diolefins Utilizing Methyl Amines as Azeotrope Formers. W. Engs, S. N. Wik, and R. M. Roberts to Shell Development Company.

A diolefin with more than 2 and less than 6 C atoms, such as butadiene or isoprene, is obtained in concentrated form from its mixture with a monoolefin which possesses a vapor pressure of the same order, such as butylenes or an amylene, respectively, by distilling the mixture in the presence of methylamine. A vapor fraction containing the major portion of the mono-olefin and a residue containing the major portion of the diolefin are formed.

U.S.P. 2,414,651. Process for the Treatment of Hydrocarbons. J. W. Latchum to Phillips Petroleum Company.

Cyclopentadiene is recovered in the form of dicyclopentadiene from an aromatic oil mixture containing the latter in admixture with monocyclic aromatic hydrocarbon and aliphatic conjugated pentadiene by heating this mixture to such a temperature and for such a period that the dicyclopentadiene is converted to the monomer. The resulting mixture, substantially free of dicyclopentadiene, is distilled in a fractionating column under such condition that the cyclopentadiene and aliphatic conjugated pentadiene are taken overhead and the monocyclic aromatic hydrocarbon is taken off in the kettle product. Steam is injected into the bottom of the column. A water layer is withdrawn from it at a point near to but above the feed entry to the column. The cooled water layer is injected into the top of the column as the sole reflux. The condensed overhead is heated under such conditions as to dimerize the cyclopentadiene without polymerizing the other unsaturated hydrocarbons present. The resulting mixture is distilled in a second fractionating column under such conditions that the aliphatic conjugated pentadiene content thereof and lighter products are taken overhead while the dicyclopentadiene is taken off in substantially pure form as the kettle product and that substantially no dedimerization of dicyclopentadiene takes place.

U.S.P. 2,414,737. Absorption of Olefins. R. L. Heinrich to Standard Oil Development Company.

An olefin-containing feed stock is passed in sequence through a first and a second absorption zone and contacted in the second zone with absorption liquid comprising H_2SO_4 and H_2O under conditions suitable for the absorption of olefins. The enriched absorption liquid from the second zone is at least partially freed from water. The thus produced concentrated rich absorption medium is employed as the absorbent in the first absorption zone. Enriched absorption liquid from the first zone is hydrolized to form alcohols, and the stock is freed from olefins.

U.S.P. 2,414,761. Separation of Hydrocarbons Having Different Degrees of Saturation. H. S. Nutting and L. H. Horsley to The Dow Chemical Company.

Aliphatic hydrocarbons which have the same boiling range and which are of different degrees of saturation and con-

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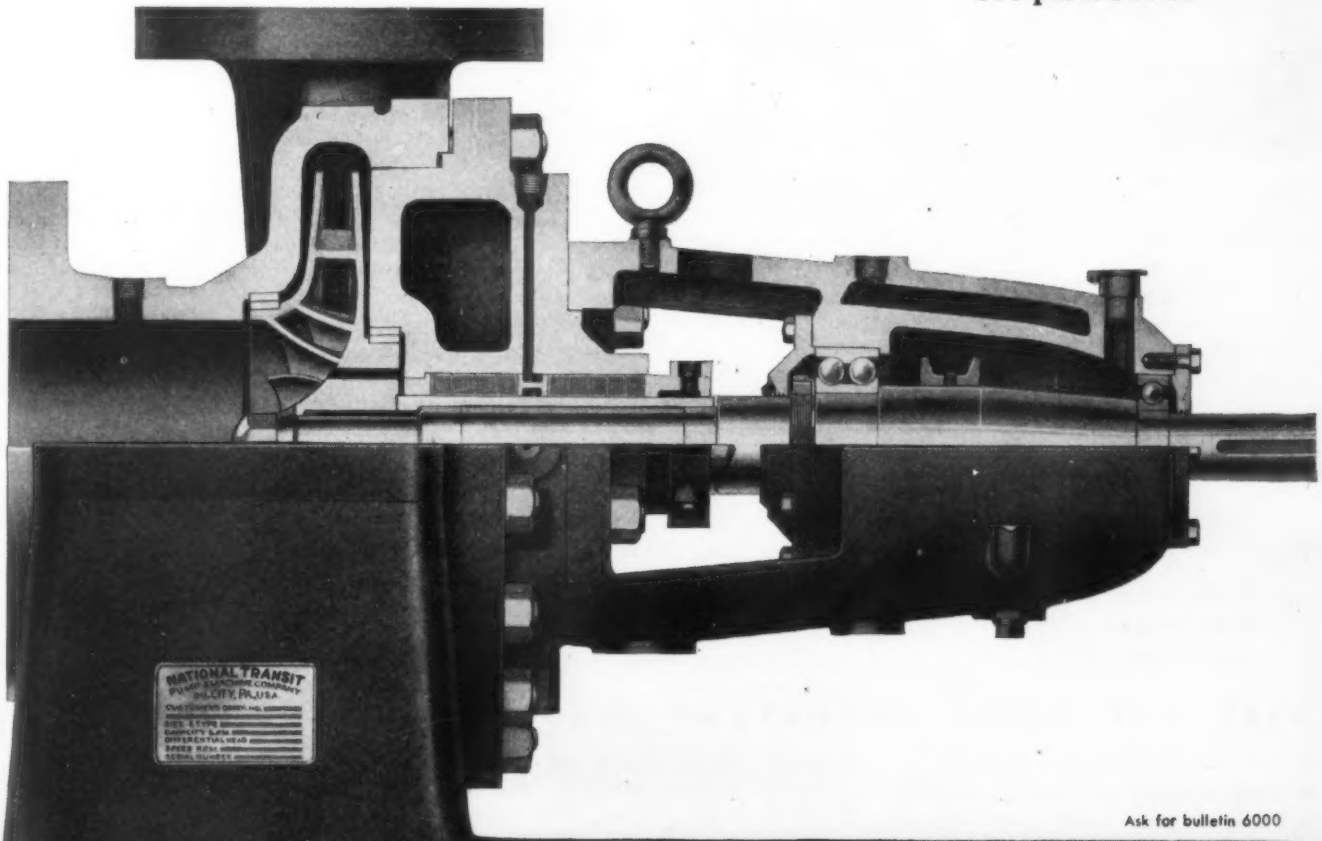

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tain more than 2 and less than 7 C atoms are separated by fractional distillation at increased pressure in the presence of at least sufficient ammonia to form an azeotrope with the more nearly saturated hydrocarbons of the mixture. An appreciable amount of water is added for the distillation in order to prevent an accumulation of solids on inner surfaces of the apparatus.

U.S.P. 2,414,817. Method of Quenching and Cooling Vapors. C. E. Kleiber, D. L. Campbell, D. E. Stines and G. T. Atkins to Standard Oil Development Company.

In a process similar to that of U. S. P. 2,414,816 the gaseous stream of reaction products containing butadiene, steam and unstable tar-forming and gum-forming impurities is treated immediately upon leaving the reaction zone by injecting water into it to cool it quickly to 900-1000° F., then cooling it by indirect heat transfer to 500-600° F., and then further cooling it by direct contact with a liquid hydrocarbon oil to about 300° F. in order to remove the tar-forming and gum-forming impurities. Finally, the reaction products are cooled to condense the steam. The process also can be used for the recovery of other diolefins obtained by catalytic dehydrogenation of more saturated hydrocarbons.

U.S.P. 2,415,006. Butadiene Separation. K. H. Hachmuth to Phillips Petroleum Company.

A mixture comprising butadiene and other closely-boiling C_4 hydrocarbons including butene-1 and butene-2 is subjected to a first fractional distillation to remove a portion of the butene-2. The remainder is extracted with a solvent selective for butadiene under conditions such that a fraction consisting essentially of butadiene and a further portion of the butene-2 is absorbed without substantial absorption of the butene-1 and the remainder of butene-2. This fraction is stripped from the solvent and then subjected to a second fractional distillation to effect separation of butadiene from butene-2. Furfural is preferably employed as the selective solvent.

U.S.P. 2,415,009. Butadiene Stabilization. L. F. Hatch, D. E. Adelson and B. O. Blackburn to Shell Development Company.

For stabilizing butadiene 0.01-0.02 percent by weight of a compound of the formula Y-X-NHR is added wherein X is aryl radical, R is hydrogen or a hydrocarbon radical, and Y is OH, NH_2 or NHR, (R, being a hydrocarbon radical). p-Amino-phenol, p-phenylen-diamine and n-benzyl-p-amino-phenol are examples of the compounds to be employed. Polymerization of butadiene by exposure to the air, corrosion of ferruginous containers by butadiene, and peroxide formation in monomeric butadiene are inhibited.

U.S.P. 2,415,161. Prevention of Corrosion. E. Q. Camp to Standard Oil Development Company.

A small amount of a low-boiling nonacidic oxygenated organic compound is incorporated into a petroleum distillate which is contacted in vapor phase at a temperature of 1300-1650° F. with an iron, chromium, and nickel alloy to inhibit corrosion. The additive preferably is employed in an amount of 1-8 percent by weight of the hydrocarbon. An aliphatic alcohol or ketone, or an ester of an

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aliphatic alcohol and an aliphatic acid are, for example, used to inhibit corrosion in refinery operations of hydrocarbons making use of heating and reaction coils manufactured from an alloy of the indicated kind.

U.S.P. 2,415,171. Method for Recovering a Substantially Olefin-Free Hydrocarbon Fraction. J. T. Horeczy to Standard Oil Development Company.

A hydrocarbon feed stock comprising olefin in an amount not in excess of 75 percent is contacted with a complex of boron halide and an alkyl or aryl monocarboxylic acid, such as a complex of BF_3 and acetic acid, at a temperature of about 160-180° F. while maintaining a ratio of 3-5 moles of acid per mol of feed stock to form a homogeneous phase. This phase is withdrawn and subjected to distillation conditions to recover a hydrocarbon fraction substantially free from olefins. Feed stocks boiling at 140-168° F. and containing 60 percent olefins and 40 percent paraffins and naphthenes, or stocks boiling at 200-250° F. and containing 30 percent olefins, 35 percent toluene, and 30 percent paraffins and naphthenes can, for example, be treated in this process.

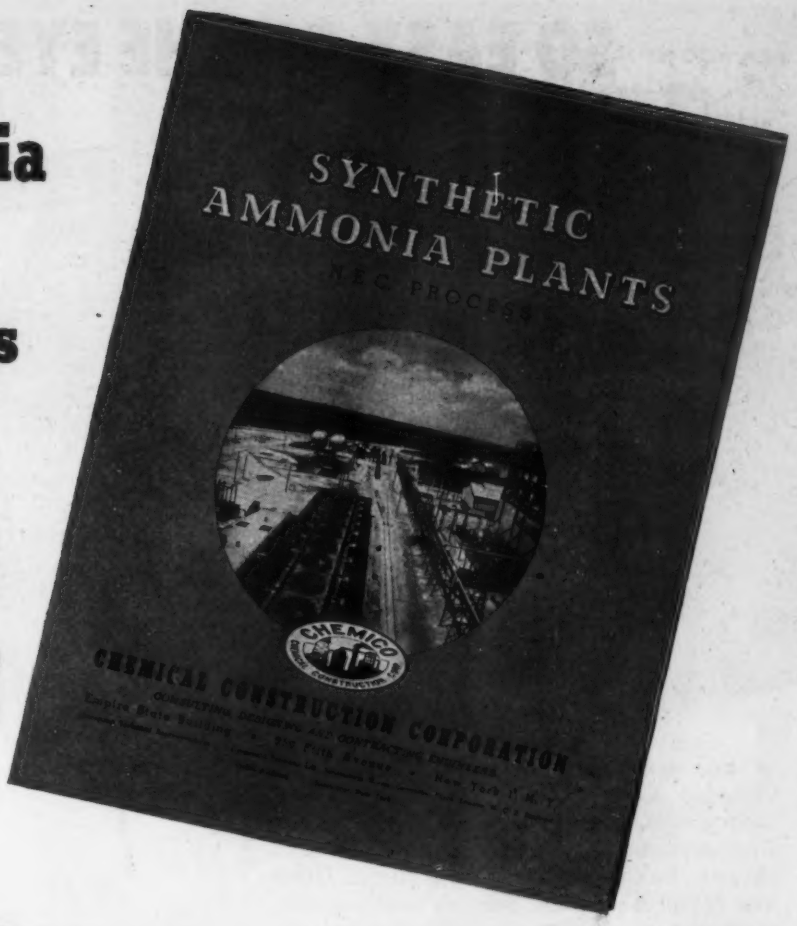
U.S.P. 2,415,192. Recovery of Azeotropic-Former in Distillation of Hydrocarbons. J. D. Rittenhouse to Union Oil Company of California.

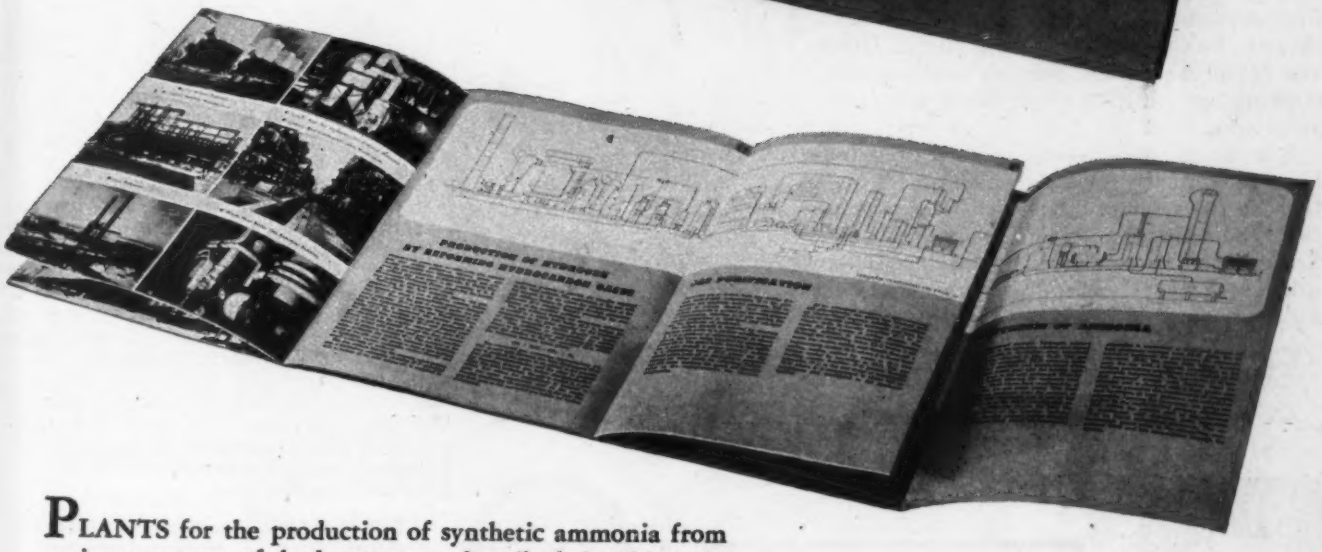
A complex hydrocarbon fraction containing chemically-similar hydrocarbon components ordinarily boiling in the same temperature range is distilled in the presence of a sufficient amount of an azeotrope-former to produce a vaporized mixture consisting of at least one of the said components together with the azeotrope-former. At least one of the other components is left in the residue. The vaporized mixture is extracted in the vapor state with a liquid selective absorbent for the azeotrope-former. The solution formed of this absorbent and azeotrope-former is separated from the hydrocarbon vapors. An organic compound which is not readily separable from the vaporized mixture by extraction with water is used as the azeotrope-former, e.g., acetone or methyl ethyl ketone; and the liquid absorbent used is a substantially non-aqueous polar organic compound other than the azeotrope-former and readily separable from the formed extract, e.g., benzyl alcohol, or a polyhydric alcohol, or an alkyl ether of the latter.

U.S.P. 2,415,337. Batch Rectification. S. C. Carney to Phillips Petroleum Company.

An oil stock is heated to distillation temperature in a batch still. The still vapors are passed into a treating zone comprising a rectifying section and a stripping section at a point intermediate both sections. In the first named section the distilled vapors are rectified to produce an overhead vaporous product and a liquid bottoms. These bottoms are passed into the stripping section where a stripper vapor and liquid stripper bottoms are produced, the bottoms being heated to facilitate the stripping section. The stripper vapor is passed in conjunction with the still vapors into the rectifying section. A portion of the stripper bottoms is recycled to the distilling charge stock as reflux, while the remainder of these bottoms is removed. The rectifier overhead vapors are condensed, and a portion of the condensate obtained is recycled as rectifier reflux while the re-

Synthetic Ammonia Production described in this *NEW* CHEMICO BULLETIN





PLANTS for the production of synthetic ammonia from various sources of hydrogen are described in this new 14-page brochure. An unusual presentation of coordinated flow charts illustrates the arrangement of equipment for the three basic types of plants. Numerous photographs clearly show the details of equipment and design.

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maining condensate is removed as product. The distillation is continued until the desired material has been distilled from the oil charge stock. The sum of the rates of heat supplied to the still and to the stripper bottoms is maintained constant throughout the distillation of a batch of oil. The process can be used for improving the octane number of natural gasoline containing n-heptane which is removed as the stripper bottoms, or for removing toluene as the stripper bottoms from a hydrocarbon stock containing it.

CATALYST PREPARATION ACTIVATION AND REGENERATION

U.S.P. 2,414,727. Recovery of Sulfuric Acid. H. L. Ellender to Standard Oil Development Company.

Sulfuric acid spent in the treatment of hydrocarbons (for instance in an alkylation process) is regenerated by subjecting an unseparated sludge acid containing previously unhydrolized hydrocarbons to hydrolysis at reduced pressure not substantially in excess of five inches of Hg.

U.S.P. 2,414,883. Catalytic Reactions. H. Z. Martin to Standard Oil Development Company.

Hydrocarbons are mixed with powdered catalyst in a reaction zone and the catalyst is maintained in a relatively dense fluidized condition therein. A purging gas is introduced to the bottom portion of the reaction zone and a confined stream of purged, spent catalyst is withdrawn from the bottom portion of the catalyst and is passed in a relatively dense condition directly to a regeneration zone disposed directly below the reaction zone and merging with it. Regenerating gas is uniformly mixed with the spent catalyst in this zone to maintain the catalyst in a fluidized turbulent condition and to regenerate it. The mixture of regenerated catalyst and gases is passed upwardly while out of contact with the confined stream of spent catalyst and is distributed uniformly and in direct contact with the body of fluidized catalyst in the reaction zone. A mixture of vaporous reaction products and regeneration gases substantially free from entrained catalyst is removed from the top of the reaction zone.

U.S.P. 2,414,884. Purification of Hydrofluoric Acid. M. P. Matuszak to Phillips Petroleum Company.

Hydrofluoric acid, when prepared by the action of concentrated H_2SO_4 on fluorspar or the like, contains small proportions of SO_2 which are undesirable when the HF is to be used as a catalyst in the conversion of hydrocarbons. The SO_2 can be removed from the anhydrous HF by treating the acid with H_2S to effect the reduction of SO_2 to S, which then is separated.

U.S.P. 2,415,003. Purification of Hydrofluoric Acid. R. C. Cole to Phillips Petroleum Company.

Sulfur dioxide contained in commercially prepared HF is removed from the latter by admixing the raw HF with at least partially spent HF which has been used in alkylating paraffins with olefins and contains a heavy olefinic oil soluble in HF. The resulting mixture is digested for a period sufficient to effect reaction between substantially all of the SO_2 and the olefinic oil to form complex sulfur-

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containing compounds. The SO_3-free HF is separated from a residual material containing the sulfur compounds.

CRACKING AND REFORMING

U.S.P. 2,414,770. Production of Diolefins by Cracking of Polymers. H. G. Schneider and V. E. Mistretta to Jasco, Inc.

Polymers with at least 10 C atoms and obtained by the polymerization of tertiary olefins are subjected to temperatures of 800-1400° F. Diolefins are obtained.

U.S.P. 2,414,973. Process for Catalytic Cracking of Hydrocarbons. E. F. Nelson to Universal Oil Products Company.

In the catalytic cracking of hydrocarbon oil the charge oil is pretreated with a minor portion of regenerated catalyst, preferably at a temperature of 800-1200° F., to remove deleterious components without effecting any substantial conversion of the oil to lower-boiling hydrocarbons. The thus treated oil is separated from the catalyst and the latter is returned to the regenerating step. The separated oil is cracked in the presence of a major portion of the regenerated catalyst. The same temperature employed in the pretreatment can be used in this cracking step or the temperature for the pretreatment can be lower than indicated before.

ISOMERIZATION

U.S.P. 2,415,061. Vapor-Phase Isomerization of Hydrocarbons. M. de Simo and F. M. McMillan to Shell Development Company.

Vapors of hydrocarbon to be isomerized are contacted under suitable conditions with a catalyst prepared by impregnating an adsorptive alumina consisting of gamma alumina with an effective amount of 15-40 percent of $AlCl_3$ or another aluminum halide.

U.S.P. 2,415,197. Process for Isomerizing Hydrocarbons. A. J. van Peski to Shell Development Company.

An isomerizable paraffinic hydrocarbon, such as butane, is continuously passed in the liquid phase through a heated zone containing solid aluminum halide which is partially dissolved in the hydrocarbon feed. The solution formed is continuously passed through a second zone where the hydrocarbon is isomerized at a moderately elevated temperature in the presence of additional solid aluminum halide. Only the liquid hydrocarbon phase containing dissolved aluminum halide is continuously passed through a third zone where precipitation of solid aluminum halide is effected by cooling.

U.S.P. 2,415,315. Isomerization of Normally Liquid Saturated Hydrocarbons. J. F. Walter and E. H. McGrew to Universal Oil Products Company.

A hydrocarbon mixture containing normally liquid paraffinic hydrocarbons is contacted with a solid adsorbent preferentially adsorbing aromatic hydrocarbons contained in the mixture. The adsorbed hydrocarbons are thus separated from the paraffins. The unadsorbed paraffins are isomerized in the presence of a metallic halide catalyst. An isomerized paraffin fraction and an uncon-

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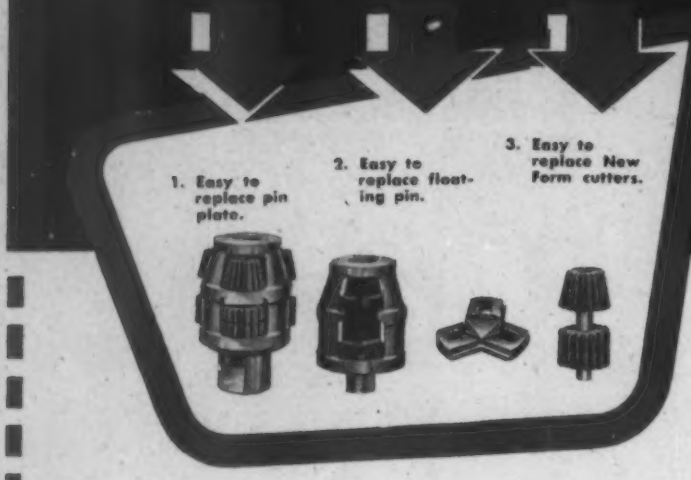

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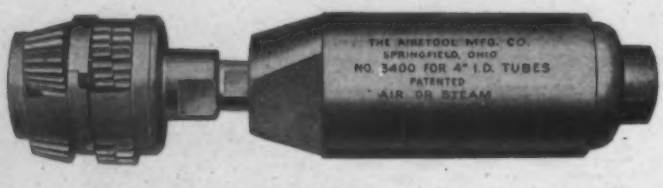

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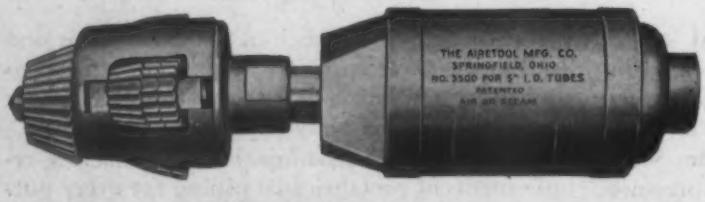

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Write for the 1947 Airetool Bulletin, No. 36, on Tube Cleaners and Expanders for Refineries. Airetool Mfg. Co., 316 S. Center St., Springfield, Ohio.

verted paraffin fraction are separated from the resultant products. At least a portion of one of these fractions is contacted with the used solid adsorbent at a temperature sufficient to remove the adsorbed aromatic hydrocarbons from the adsorbent. If the fraction of isomerized paraffins is employed for removing the aromatic hydrocarbons from the adsorbent, a mixture of aromatic hydrocarbons and isomerized paraffins is obtained which can be used as a hydrocarbon blend suitable as aviation fuel.

ALKYLATION

U.S.P. 2,414,271. Catalytic Alkylation. A. A. O'Kelly, J. R. Meadow, and R. E. Woodward to Socony-Vacuum Oil Company

For the production of high-antiknock motor fuel components, ethylene is reacted with an isoparaffin, such as isobutane, in the presence of HF at a temperature of 700-900° F. A pressure of 500-5000 psi may be employed in the reaction, but the conditions shall be such that all the reactants are in the vapor phase.

U.S.P. 2,414,764. Method for Separating Tertiary Olefins. R. F. Pfennig to Standard Oil Development Company.

A hydrocarbon feed stock containing tertiary olefin is contacted in admixture with phenol with an alkylation catalyst under conditions to cause the alkylation of a substantial portion of the tertiary olefin with phenol. A resulting liquid fraction which includes alkylated phenol and hydrocarbon is distilled to separate at least a major portion of the hydrocarbon from the alkylated phenol. A portion of the alkylated phenol fraction formed is returned to the reaction zone while another portion of this fraction is dealkylated to recover tertiary olefin. Isobutylene can, for example, be isolated by this process.

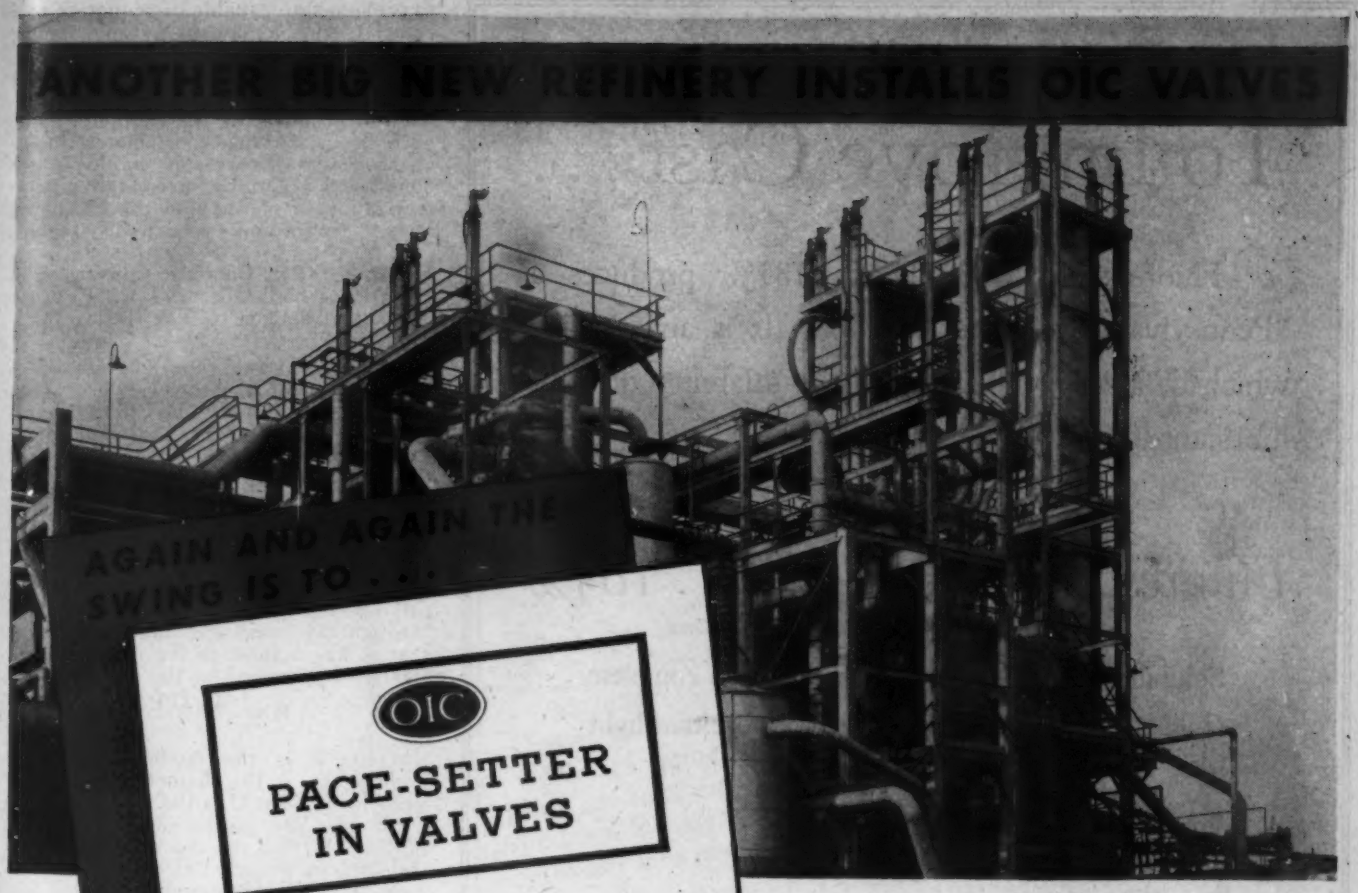
POLYMERIZATION AND CONVERSION

U.S.P. 2,414,205. Production of Motor Fuel. E. H. Lang to The Pure Oil Company.

Hydrocarbon oil is first converted into hydrocarbons rich in olefins boiling within the gasoline boiling range and into hydrocarbon gases containing 1-4 C atoms. The C_1 and C_2 gases then are chlorinated to form alkyl chlorides, which in turn are used to alkylate the higher-boiling portion of the hydrocarbons boiling in the gasoline boiling range in the presence of $AlCl_3$. The resulting alkylate is blended with the lower-boiling gasoline fraction rich in olefins to produce motor fuel.

U.S.P. 2,414,206. Conversion of Hydrocarbons. E. T. Layng to The Polymerization Process Corporation.

Olefin hydrocarbons to be employed in conversion reactions are activated by passing them under suitable reaction conditions of temperature, pressure and space velocity through a consolidated granular mass consisting of an intimate mixture of catalyst granules and spacer granules. The catalyst granules are in physical non-adhering contact with the spacer granules and comprise, each of them, an intimate mixture of a metal



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pyrophosphate capable of reduction to a catalyst, such as copper pyrophosphate, and finely divided supporting material. Each of the spacer granules is composed of a material non-reactive with the reduction products of the pyrophosphate, such as a carbonaceous material like charcoal. The process is employed for conversion reactions such as the polymerization of olefins and the alkylation of iso-paraffin and cyclic hydrocarbons.

U.S.P. 2,414,256. Catalytic Conversion of Hydrocarbon Oils. duBois Eastman and C. Richker to The Texas Company.

A preheated and vaporized stream of a charge stock of heavier hydrocarbon oil with a C residue of less than 0.2 percent and a color of less than 200 on the Lovibond half-inch scale is passed through an alumina-silica-zirconia catalyst mass containing about 20 percent Al_2O_3, 70 percent SiO_2, and 5 percent ZrO_2. This mass is maintained at a temperature of 800-1000° F. and under a pressure between atmospheric and 100 psi. The conditions of hydrocarbon flow through the mass are maintained such that R has a value in the range of 100-1000 as determined by the equation:

$$R = \frac{D_p U P}{Z}$$

wherein R is the modified Reynolds number, D_p is the diameter of catalyst particles in feet, U is the average velocity in feet per second of fluid mixture flowing through the reaction chamber if the latter is regarded as empty, P is the average density in pounds per cubic foot of fluid mixture in the reaction chamber under the operating conditions of temperature and pressure, and Z is the viscosity of the fluid mixture in the reaction chamber in pounds per foot per second under the same conditions. The flow of hydrocarbons through the mass is continued without reactivation of the latter for at least several hours. A substantial naphtha yield with a C yield of not more than 0.55 percent by weight of the feed oil is obtained. The catalyst is reactivated in situ.

U.S.P. 2,414,328. Method of Operating Polymerization Plants. R. D. Pinkerton to Sinclair Refining Company.

In order to produce polymerized olefins a gaseous mixture containing both butylenes and propylene is contacted with a catalyst, and the products obtained are stabilized in a rectifying zone by separating them into a bottoms fraction containing polymerized olefins substantially free from hydrocarbons with less than 4 C atoms, an overhead fraction containing only a minor proportion of hydrocarbons with more than 3 C atoms, and an intermediate fraction containing at least a substantial proportion of butylenes. The ratio of butylenes to propylene in this intermediate fraction shall substantially exceed the corresponding ratio in the composite gaseous mixture employed as the starting material. This intermediate fraction is recycled to the catalyzing operation. A motor fuel of high antiknock value is obtained.

U.S.P. 2,414,373. Conversion of Fluid Reactants. C. G. Gerhold to Universal Oil Products Company.

Hydrocarbons are contacted at conversion temperature with subdivided solid catalyst in a reaction zone. The con-

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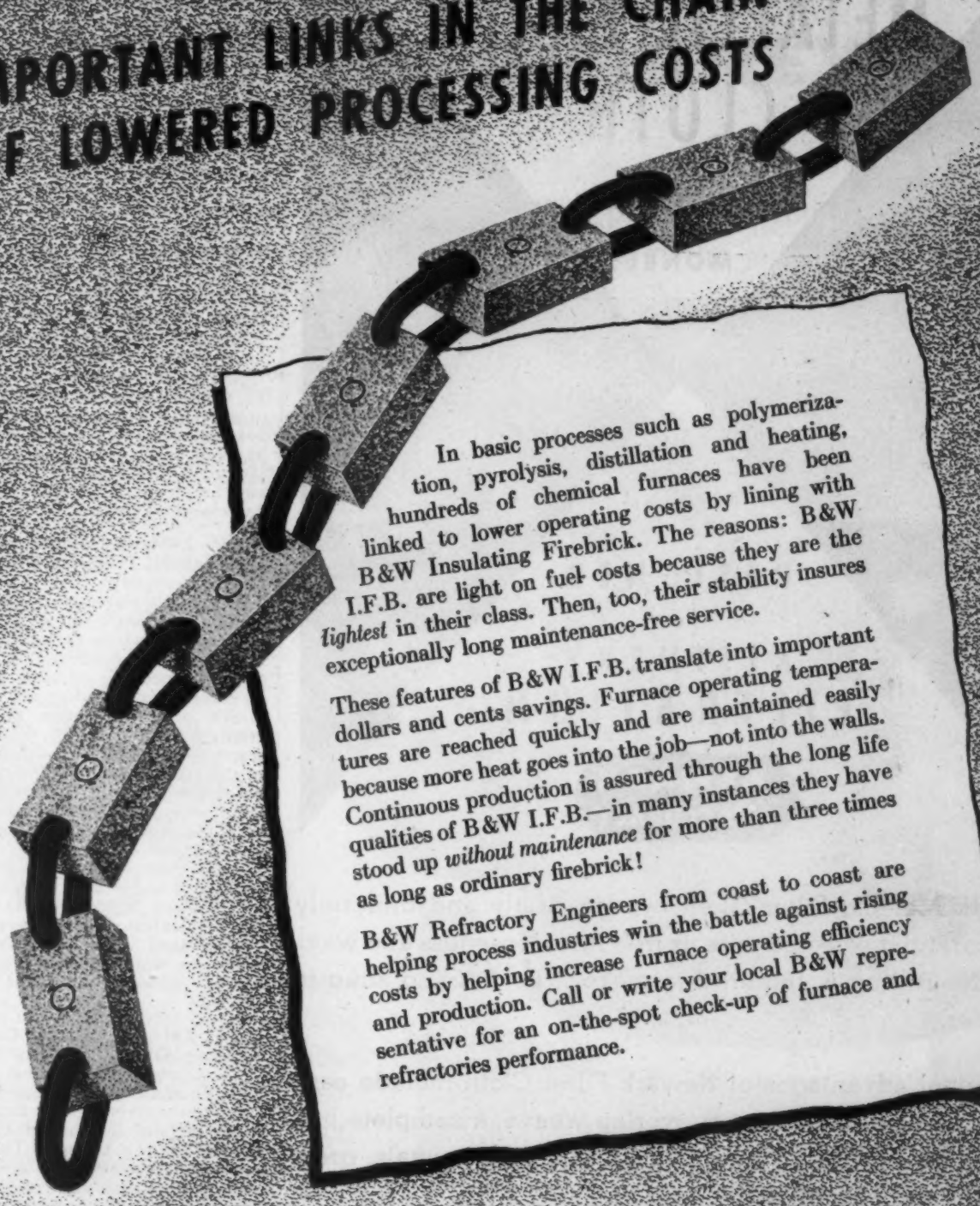

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taminated catalyst particles are removed from this zone and regenerated by contact with an oxygen-containing gas. The resultant combustion gases and regenerated catalyst are separately removed from the regenerating zone. The regenerated particles then are suspended in at least a portion of the combustion gases, and this suspension is passed through a cooling zone. The cooled suspension is introduced into a separating zone disposed at a higher elevation than the reaction zone. A portion of the cooled regenerated catalyst separated from the combustion gases in the separating zone is supplied directly from this zone to the regenerating zone without prior passage through the reaction zone while another portion of this catalyst is passed by gravity to the reaction zone.

U.S.P. 2,414,380. Polymerization of Hydrocarbons. C. B. Linn to Universal Oil Products Company.

An olefinic hydrocarbon with at least 3 C atoms is polymerized in the presence of a liquid HF catalyst containing a minor proportion of HCN. Normally liquid hydrocarbons boiling in the gasoline range are thus obtained from propylene and butylenes. The HCN serves as a moderator of the reaction.

U.S.P. 2,414,646. Hydration of Cyclic Olefins. H. J. Hepp to Phillips Petroleum Company.

A partially-water-soluble cycloalkanol containing a single ring of not more than 6 C atoms, such as cyclopentanol or cyclohexanol, is produced by absorbing the corresponding cycloalkene (e.g. cyclopentene or cyclohexene) in aqueous H_2SO_4 of, for example, 60-80 percent strength. The mixture is diluted with water recovered from a subsequent stage of the process and containing cycloalkanol. Unreacted cycloalkene is recovered, and the cycloalkanol formed is separated from the aqueous acid by distillation, the cycloalkanol distilling as an azeotropic mixture with water. The resulting cycloalkanol-water azeotrope is condensed and allowed to stratify into two layers. The cycloalkanol is recovered from the cycloalkanol layer containing water while the water layer containing cycloalkanol is recycled for the dilution of the acid.

U.S.P. 2,414,736. Catalytic Conversion of Heavy Oils. J. B. Gray to Standard Oil Company of Indiana.

Salt-contaminated heavy hydrocarbon oils are converted by the action of solid, finely divided refractory catalyst suspended in the oil vapors. The catalyst is continuously cycled from a reaction zone to a regeneration zone and back again. A minor aliquot proportion (e.g. 0.5-10 percent) of the regenerated catalyst is withdrawn from the regeneration zone, washed with water to remove salt therefrom, and then passed to the conversion zone. Lighter products including gasoline free of salts are obtained.

U.S.P. 2,414,760. Selective Polymerization of Mono-Olefins. H. O. Mottern to Standard Oil Development Company.

Tertiary C_4-C_8 olefins, such as isobutylene, contained in a refinery C_4-C_8 fraction which also contains normal C_4-C_8 olefins, are selectively polymerized in a two-stage process. In the first stage tertiary olefin is selectively extracted by intimate contact with an aqueous benzene sulfonic acid solution of 50-75 percent concentration at a temperature of

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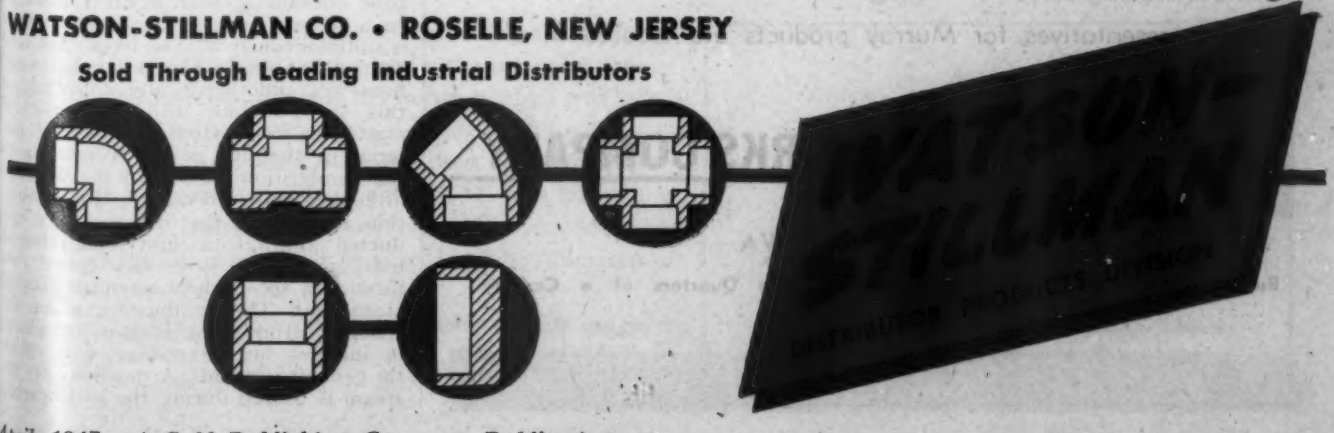

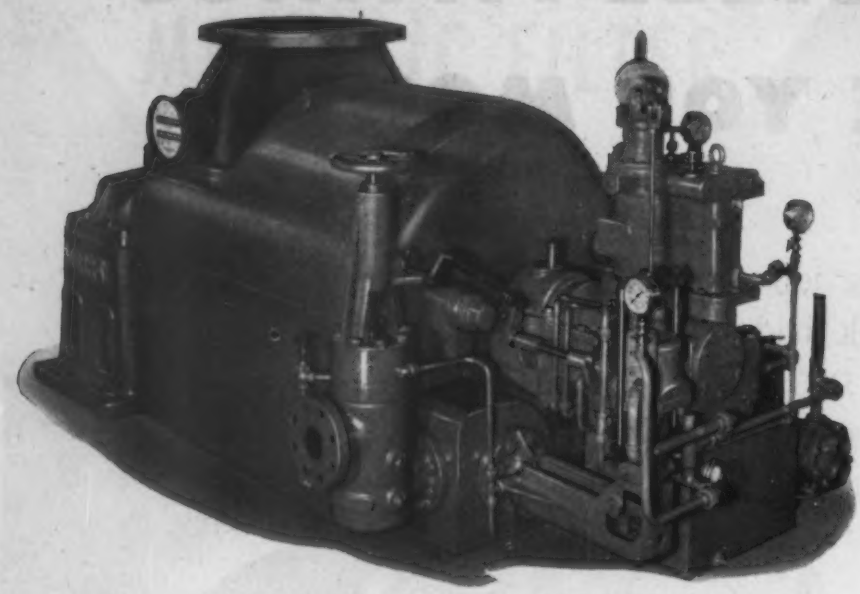

One of two MURRAY multistage steam turbines, designed to deliver 685 horsepower on steam at 110 pounds and exhausting to 26½ inches Hg vacuum, furnished to drive high speed centrifugal compressors for refrigeration in one of the most prominent Southern Hotels. A third unit has recently been shipped. Equipment includes variable speed oil relay governor, trip and throttle valve, strainer surrounding the double seated balanced governor valve, and force feed lubrication.

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40-65° C. for a period of 20-60 minutes. In the second stage, directly following the first stage, the extract of tertiary olefin is subjected to a temperature of 100-140° C. under increased pressure (e.g. a pressure of 105 psi), whereby tertiary olefin polymer is formed.

U.S.P. 2,414,812. Catalysis. E. J. Houdry to Houdry Process Corporation.

Catalytic reactions are effected in a cycle of alternating endothermic and exothermic regeneration operations by contacting hydrocarbons (at controlled temperatures above 700° F.) with a contact mass consisting of discrete pieces of active catalytic material uniformly mixed with discrete pieces of a relatively inactive heat-absorbing material of a density of at least 3 and capable of withstanding temperatures of about 2000° F. The inactive heat-absorbing material preferably shall have a size sufficiently differing from that of the catalytic material to permit segregation by screening, and the volumetric ratio of the active material to the inactive material in the contact mass is within the range of 1:5 to 3:1. Fused alumina can be used as the inactive material.

U.S.P. 2,414,852. Catalytic Process and Apparatus. H. E. W. Burnside and H. J. Ogorzaly to Standard Oil Development Company.

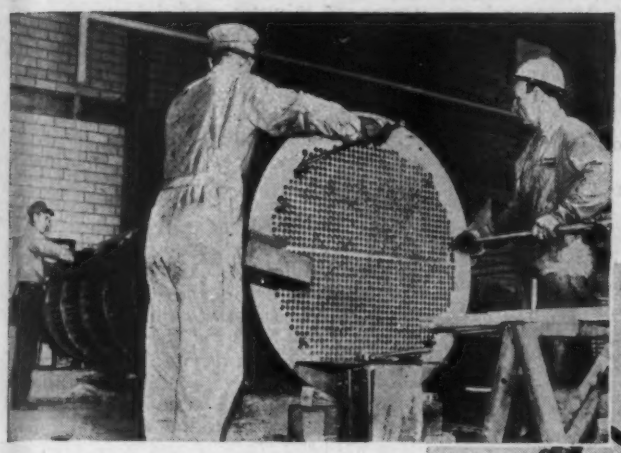
In the catalytic conversion of hydrocarbon oils, particularly in catalytic cracking, finely-divided catalyst is continuously circulated through a conversion zone and a regenerating zone and the catalyst is maintained in a relatively dense fluidized layer within the conversion zone. The hydrocarbons are passed upwards through this zone at a rate controlled to prevent entrainment of the catalyst in the hydrocarbon vapors. A second batch of the catalyst is maintained in the regeneration zone located at a substantially different elevation than the conversion zone, and a confined vertical column of the catalyst is maintained between these zones and is open to both zones. The catalyst is maintained in a freely-flowing, fluidized state capable of generating a fluid pressure within said column. A stream of the catalyst is continuously withdrawn from the lower of the two zones and is discharged at a controlled rate into the stream of gases passing to the upper zone. The back pressure on the lower treating zone is controlled to regulate the level of catalyst in the upper zone.

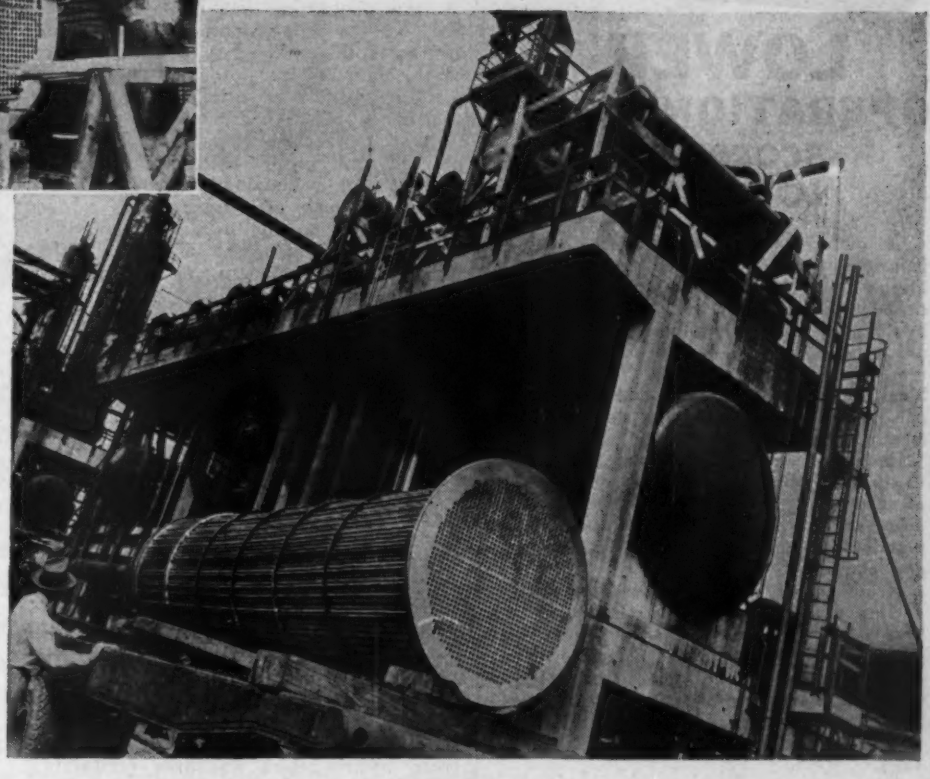
U.S.P. 2,414,972. Conversion of Hydrocarbons. E. F. Nelson to Universal Oil Products Company.

Hydrocarbons are subjected to endothermic conversion in a first reaction zone containing solid contact material. In a second zone carbonaceous matter is simultaneously burned from such solid contact material. Thereafter hydrocarbons are endothermically converted in this second zone while carbonaceous matter is burnt from the contact material in the first zone. Admixture of the combustion gases and the products of conversion is prevented. Hot combustion gases from the first zone are conducted through a first heat-retentive mass and those from the second zone through a second heat-retentive mass to store heat. The combustion gases discharging from these masses are passed in indirect heat exchange with water to generate steam. A portion of this steam is passed during the hydrocarbon

Light Hydrocarbon from Fractionating Towers

handled by ANACONDA ARSENICAL ADMIRALTY TUBES...





(Above) Inserting Anaconda Arsenical Admiralty 439 Tubes in tube bundle at Houston, Texas, butadiene plant of Sinclair Rubber, Inc. Photograph at right shows tube bundle ready for installation in one of the shells on top of superstructure.

THE CONDENSERS shown in the photographs above are located at the butadiene plant of Sinclair Rubber, Inc., Houston, Texas. They were built to handle light hydrocarbon from the fractionating towers.

In order to provide the required heat transfer and economical service under the corrosive conditions encountered, the tubes being installed are Anaconda Arsenical Admiralty 439.

This Anaconda Alloy has been manufactured on a commercial basis since 1934. It is but one of 11 standard and several special condenser tube alloys currently produced by The American Brass Company.

This list provides tubes to meet a wide range of heat transfer requirements. The broad metallurgical knowledge and practical experience of our Technical Department is available in helping you make the right selection. 48174

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conversion through the contact mass in the first zone and commingled there with the hydrocarbons. Another portion of the steam is correspondingly passed through a second zone during the conversion of hydrocarbons therein. Normally-liquid, relatively light hydrocarbons, such as naphtha, gasoil and the like, or normally-gaseous hydrocarbons, such as butanes and butylenes, can be converted by means of this process to more valuable normally-gaseous fractions, containing butadiene, acetylene, etc. In the cracking of naphtha temperatures of 1400-1600° F. can, for example, be used for a period of 0.1 second or less, while in the catalytic conversion of butane and butylene temperatures of 1050-1150° F. can be used for the same period under subatmospheric pressure.

U.S.P. 2,415,101. Method for Making Phenol. R. H. Krieble and W. I. Denton to Socony-Vacuum Oil Company, Inc.

A mixture of crude-benzol vapor and oxygen containing gas is passed under pressure through a heated reaction zone void of solid catalyst to convert a part of the benzene in the crude benzol to phenol and other oxidation products, such as diphenyl. Unreacted substantially-pure benzene is separated from the phenol and other oxidation products formed and is recycled in the reaction mixture. Fresh crude benzol is added continuously to the unreacted substantially-pure benzene at a greater rate than that at which benzene is converted to phenol and the like. Excess pure benzene is removed prior to its admixture with the fresh crude benzol containing one or several of the usual impurities, viz. a paraffin, an olefin, and cyclohexene. The addition of the fresh crude benzol and the removal of the excess pure benzene is so proportioned that a concentration of 0.25-5 percent of said impurities is maintained in the mixture.

U.S.P. 2,415,102. Process for the Preparation of Pure Oxygen Containing Derivatives of Aliphatic Hydrocarbons. A. Landgraf and O. Roelen, vested in the Alien Property Custodian.

Pure oxo compounds of alkene hydrocarbons, including aldehydes, alcohols, fatty acids, and ketones, are prepared from hydrocarbon mixtures containing alkenes by separating the hydrocarbon mixtures into alkene fractions containing molecules the C atoms of which differ in number from two to three. The alkenes are subjected to catalytic action with CO and H until the number of C atoms to the molecule has been raised by one. The boiling range for each of the fractions containing oxo compounds is thereby raised so that it is much higher than the highest boiling point of the corresponding alkene fraction of the original mixture. The catalyzer is removed from each fraction, the aldehydes formed are reduced to alcohols, and the latter are obtained in a pure state by vacuum fractionation. The separation of the charge hydrocarbon mixture by fractionation is, for example, carried out in such a manner that the first fraction C_{10}-C_{13} alkenes boils at 165-220° C., the second fraction C_{12}-C_{14} alkenes at 200-225° C., the third fraction C_{13}-C_{16} alkenes at 255-291° C., and the fourth fraction C_{17}-C_{20} alkenes at 295-350° C.

U.S.P. 2,415,172. Production of Polymers from Alcohols Using a Boron Fluoride-Acetic Acid Complex Catalyst. J. T. Horeczy to Standard Oil Development Company.

An aliphatic alcohol containing a tertiary C atom is contacted with a complex of BF_3 and acetic acid for a sufficient interval to form a high-boiling hydrocarbon polymer. Tertiary butyl alcohol is, for example, treated to form a high-boiling hydrocarbon suitable for use in a high-octane motor fuel. 5-40 percent of the catalyst complex can consist of BF_3. The contact time can vary between 30 minutes and 2 hours.

U.S.P. 2,415,272. Conversion of Hydrocarbons. W. L. Benedict and W. J. Mattox to Universal Oil Products Company.

Gasoline and alkyl aromatic hydrocarbons are produced in combination by cracking a fresh hydrocarbon oil (e.g. a Gulf Coast gasoil) and an aliphatic recycle stock and separating from the resultant product a gasoline fraction with an end point of 250-350° F. and a naphtha fraction boiling above the range of the gasoline fraction and having an end point of 400-500° F. The olefins present in the naphtha fraction are converted to more saturated hydrocarbons, and an olefin-free naphtha section is separated. Benzene is commingled with this olefin-free naphtha fraction and is reacted with poly-alkylated benzene contained in this fraction to effect the transfer of alkyl groups to the benzene. Unconverted aliphatic hydrocarbons and an olefin-free mono-alkylated benzene are separated from the products of the last-named step. The aliphatic hydrocarbons are recycled to the cracking step as the above mentioned recycle stock, while mono-alkylated benzene is recovered. Toluene or ethyl benzene, for example, are produced by the process. A silica-containing catalyst can be employed in the cracking step as well as in the reaction of benzene with poly-alkylated benzene.

HYDROGENATION
DEHYDROGENATION
AROMATIZATION

U.S.P. 2,414,276. Catalytic Conversion Process. E. E. Sensel and R. A. Beck to The Texas Company.

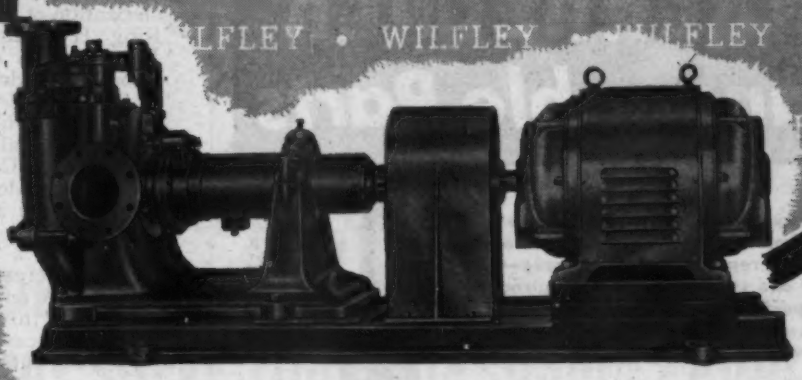
Compounds of 2 or more C atoms are formed by continuous hydrogenation of oxides of carbon in the presence of a powdered catalyst comprising about 64 percent diatomaceous earth, 32 percent cobalt, and 4 percent thorium and magnesium oxides. A mixture of an oxide of carbon and hydrogen is continuously passed over this catalyst under conditions such that normally gaseous and normally liquid compounds are formed, e.g., at a temperature of 320-420° F. Reaction products and catalyst are continuously withdrawn from the reaction zone. A naphtha fraction and a gasoil fraction are separated from the reaction products. The spent catalyst is treated with a portion of the gasoil at a temperature of 500-800° F. in the presence of hydrogen, whereby the gasoil is cracked and the catalyst is reactivated.

U.S.P. 2,414,685. Catalytic Dehydrogenation. F. T. Eggertsen and H. H. Voge to Shell Development Company.

A hydrocarbon is continuously dehydrogenated in the presence of steam

Cost-Saving

Performance



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150 lb. S. P.
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Lunkenheimer part-interchangeability is fully illustrated and described in Circular No. 567.

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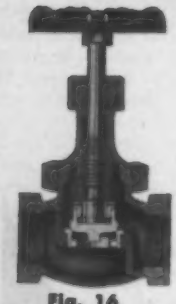

Fig. 16
Bronze Regular
"RENEWO"

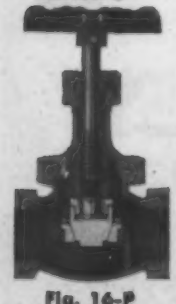

Fig. 16-P
Bronze Plug Type
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There are fewer parts to be carried, repairs are more quickly and easily made. The labor, time, and expense of valve maintenance is cut down—'way down!

SAVE THIS WAY, TOO!

For severe throttling service, a regular type can be converted into a plug type, by simply changing the seat and disc, without taking the valve out of the line.

Thus it's not necessary to buy a whole new valve—just a matched set of plug type seat and disc which interchange perfectly in corresponding patterns and sizes in a range of pressures from 150 to 350 lb. S. P.

Lunkenheimer simplicity of design, minimum number of parts, and fine precision workmanship make this and other outstanding economies possible.

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with a catalyst consisting of magnetic oxide of iron and potassium carbonate prepared by combining a powdered iron oxide which has been calcined at a temperature of 700-950° C. for a sufficient time to reduce the available surface to below 8 square meters per gram with from 25-100 mol percent (calculated as K_2O and based on the iron oxide calculated as Fe_3O_4) of a compound of K convertible to potassium carbonate under the reaction conditions. The catalyst mixture obtained is formed into pellets which are hardened by heating at a temperature below 800° C.

U.S.P. 2,414,620. Catalytic Dehydroisomerization of Five-Membered Alicyclic Ring Hydrocarbons Having Six or More Carbon Atoms per Molecule to Aromatic Hydrocarbons. R. A. Trimble to Shell Development Company.

A mixture of hydrocarbons containing five-membered alicyclic ring compounds with at least 6 C atoms to the molecule is contacted under dehydroisomerization conditions with a catalyst which essentially consists of molybdenum sulfide and contains a promoting amount of about 2-15 percent of nickel in the sulfide state. Aromatic hydrocarbons are obtained by this process, e.g., benzene from a hydrocarbon mixture containing methylcyclopentane.

U.S.P. 2,414,816. Method for Dehydrogenating Normal Butenes to Form Butadiene. C. E. Kleiber, D. L. Campbell, D. E. Stines and C. C. Nelson to Standard Oil Development Company.

N-butenes are heated to a temperature somewhat below reaction temperature while steam is separately heated to a temperature above that of the reaction. The heated materials are mixed and immediately injected into a reaction zone where the butenes contact a dehydrogenation catalyst at a reaction temperature of 1100-1300° F. The reaction products are withdrawn from the reaction zone after a relatively short period to prevent undesired reactions, and then are quenched immediately to temperatures below 1000° F. They are further cooled to condense at least a major portion of the steam while the butadiene formed is left in vapor phase. The uncondensed portion of the reaction products is cooled and fractionated to recover butadiene therefrom. The steam and other diluent to butene ratio employed may vary from 7-15. Water may be injected for quenching the reaction products leaving the reaction zone.

U.S.P. 2,414,889. Destructive Hydrogenation. E. V. Murphree to Standard Catalytic Company.

Lower-boiling hydrocarbons suitable as motor fuel are obtained from a higher-boiling hydrocarbon oil of the gasoil type by treating the latter with 5000-20,000 cubic feet per barrel of oil of a mixture of hydrogen and methane containing the methane in a proportion of 40-85 mol percent. The treatment is carried out at a temperature of 500-1000° F. under a pressure of 60-1000 atmospheres and in the presence of a catalyst comprising a minor proportion of a sulfide of a metal of group VI of the periodic system and a major proportion of a clay pretreated by HF. A substantial proportion of the methane reacts with the oil. The products of the treatment are cooled without reduction of pressure. Liquid and gaseous products are separated and the pressure on the liquid products then is reduced

Photo: Courtesy of Henry Vogt Machine Co., Louisville, Ky.

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No one metal or alloy can be expected to withstand the many different corrosive elements encountered in chemical plants, petroleum refineries and food processing industries. Bridgeport has developed a number of condenser tube alloys designed to meet a wide range of operating conditions. Selection of the tubing best suited for a particular application requires considerable experience and knowledge. Bridgeport maintains a consistent program of corrosion research involving laboratory work as well as field and service tests. Contact the nearest Bridgeport office for technical service to help reduce premature tube failures, and write for 112-page Condenser Tube Manual.

ARSENICAL ADMIRALTY—for handling sea water or polluted fresh circulating water, hydrocarbon gases containing sulfur compounds, lubricating oils, gasoline, naphtha, etc.

ARSENICAL MUNTZ*—excellent for resisting high sulfur compounds as well as dezincification from corrosive water. More effective than ordinary Muntz.

CUZINAL—superior to Admiralty for resisting air impingement corrosion or erosion-corrosion produced by high water velocities. Resists dezincification and sulphide corrosion encountered in condensers and coolers handling various types of petroleum products.

CUPRO NICKEL—resists stress corrosion cracking caused by moist ammonia, oxygen and stress. Shows good resistance to caustic and other alkaline liquids and salt solution. Fine resistance to air impingement corrosion produced by high sea water velocities.

RED BRASS—resists corrosive waters and hydrocarbon vapors. Better for resisting stress corrosion than Admiralty or Cuzinal, but not recommended for severe sulfur corrosion.

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DUPLEX TUBING—for double corrosive conditions too severe for single metal or alloy—extensively used in ammonia refrigeration systems and heat exchangers and condensers handling a wide variety of organic and inorganic chemicals. Available in Red Brass, Copper, Cuzinal, Cupro Nickel, Muntz, and Duronze IV ... with steel, aluminum, stainless or monel. Inner and outer tubes are selected to combat a specific corrosive element and mechanically combined to give good heat transfer. Our Technical Bulletin No. 746 covers applications and methods of installing Duplex Tubing.

*U. S. Pat. No. 2,118,688 **U. S. Pat. No. 2,082,380

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Condenser and Heat Exchanger Tubing

BRIDGEPORT BRASS

so that a portion thereof is vaporized. The still liquid products are fractionated to obtain a fraction boiling in the motor-fuel range and a higher-boiling fraction. The vapors evolved by the reduction of pressure are combined with the gaseous products separated before and the vapor mixture is recycled to the reaction to provide at least a portion of the mixture of hydrogen and methane.

U.S.P. 2,414,951. Application of Metal Sulfide Catalysts in Vapor-Phase Treatment of Mineral Oils. Z. V. Jasaitis and D. D. Davidson to Shell Development Company.

In treating mineral oils in the vapor phase with a metal sulfide catalyst the following steps are employed in repeated sequence: (1) dehydrogenation at a temperature above 800° F. of mineral oil containing less than 0.10 percent S to increase the aromatic content of the oil; (2) removal of carbonaceous deposits from the catalyst by burning; (3) desulfurization of the obtained mineral oil containing at least 3 percent S under hydrogenation conditions at a temperature below 800° F. Nickel sulfide or cobalt sulfide, if desired, in combination with tungsten sulfide or molybdenum sulfide, can be employed as the catalyst.

U.S.P. 2,414,962. Manufacture of Butadienes. W. J. Mattox to Universal Oil Products Company.

A mixture of n-butane and n-butene is dehydrogenated to form butadiene which is separated from the conversion products containing a minor amount of isobutene and lower-boiling gaseous products incidentally formed. The isobutene and the gaseous products are separated from the remaining products and only the latter are returned to the dehydrogenation step.

U.S.P. 2,415,065, 2,415,066. Cycloparaffin Production. W. E. Ross and P. Pezzaglia to Shell Development Company.

Cyclohexane is obtained from a naphthenic gasoline fraction comprising methylcyclopentane, cyclohexane, and open-chain paraffins of about the same boiling range by contacting this fraction with a Friedel-Crafts metal halide isomerization catalyst under methylcyclopentane isomerizing conditions. The resulting isomerizate is thermally cracked at a temperature of 550-700° C. for 5-30 seconds. The open-chain paraffins are thereby selectively converted to lower-boiling hydrocarbons. Hydrocarbons comprising cyclohexane and hydrocarbons comprising methylcyclopentane are separated from the cracked products by fractional distillation, and the hydrocarbons comprising methylcyclopentane are recycled to the isomerization step.

According to U. S. P. 2,415,066, the starting mixture comprising methylcyclopentane, cylohexane and open-chain paraffins with 6 and 7 C atoms is first treated to separate a lower-boiling fraction comprising methylcyclopentane and a higher-boiling fraction comprising cyclohexane, methylcyclopentane and open-chain paraffins with 7 C atoms. The lower-boiling fraction is isomerized in the above indicated manner, and the higher-boiling fraction is thermally cracked as indicated before and for the indicated purpose.

DESULFURIZATION

U.S.P. 2,414,626. Desulfurization in Hydrocarbon Conversions. J. G. Allen to Phillips Petroleum Company.

In the alkylation of hydrocarbons containing organic sulfur compounds in the presence of an HF catalyst, acid-soluble oils formed during such conversion of hydrocarbons are added to the reactants. These oils absorb the sulfur compounds. They are separated from the effluent of the reaction zone. At least a portion of the acid-soluble oils in desulfurized and recycled to the reaction zone.

U.S.P. 2,414,963. Process of Removing Sulphur from Mineral Oil. E. B. McConnell to The Standard Oil Company (Ohio).

Sulphur compounds contained in crude oil are selectively heat-cracked at a temperature just below oil-cracking temperature, preferably above 500° F. The temperature of the oil is lowered and the hydrogen sulfide formed is distilled off. The oil subsequently is fractionated.

HEAVY OILS AND WAXES

Re. 22,829, 22,830. Compounded Hydrocarbon Oil (and Compounded Oil, Respectively). J. T. Rutherford and R. J. Miller to California Research Corporation.

The hydrocarbon oil composition described which is to be used particularly as a lubricant comprises a major proportion of hydrocarbon oil of lubricating viscosity and a small amount of a polyvalent metal salt of a sulfur-containing acid of P having at least one oil-solubil-

J-M Interlocked No. 270 SEALS HOT OIL with less gland pressure

Less gland pressure means more life for packing! That's one of the reasons why J-M No. 270 Interlocked lasts longer in hot oil service.

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Have you reduced HCl corrosion to a minimum?	☐	☐
Are maintenance costs low?	☐	☐
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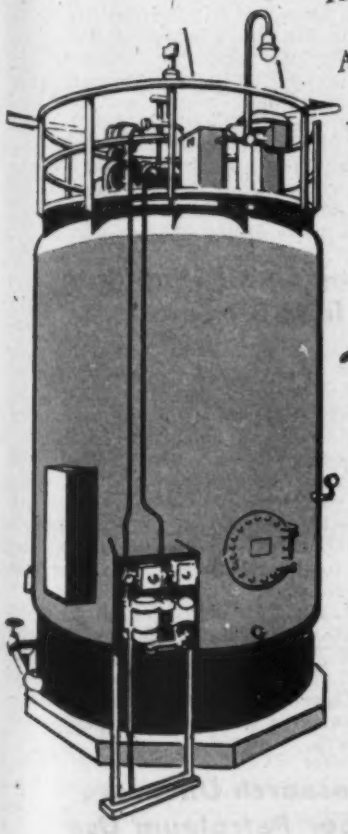
Are you getting maximum continuous time on stream?	☐	☐
Can you operate on a time schedule?	☐	☐



...if not

get the facts about Petreco Desalting to help your refinery.

The Benefits of Petreco Desalting go far beyond efficient salt removal. Each refiner benefits in his own particular way but *every* refiner reports *more on stream time, reduced corrosion* and a *smoother operating schedule.* Many have simplified crude tank bottoms disposal by merely adding "the bottoms" to the crude entering their Petreco Desalters. Others show substantial economies by taking crude oils best suited for manufactured products and under most favorable price quotations and desalting them, rather than by restricting purchases to low salt content crudes in order to conform with the salt tolerance of their refining equipment. No experienced operator can overlook the importance of a *time schedule.* With properly desalted crudes, shut downs are *scheduled* according to a planned routine; — they don't just happen when the equipment "plugs up"!

Why miss any good bets? It costs you nothing to have a talk with a Petreco Engineer. It costs you nothing to have Petreco check up on the salt situation in your refinery. It costs you nothing to have all the facts . . . and you stand to make money on the deal! *Plenty of refiners have.* Plan with Petreco for desalting profits.

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PETRECO

Electric **DESALTING ★ DEHYDRATING**

PR 47-D

izing organic substituent and containing at least 24 C atoms. Enough of the salt is added to stabilize the oil against deterioration by heat and oxidation. Between 0.25 and 2 percent by weight of a salt of an acid ester produced by a reaction of the type:

$$2\,ROH + P_2S_5 \rightarrow R_2PO_2S_2H + H_2S$$

is, for example, used, wherein ROH is an alcoholic or phenolic hydroxy compound, the Cr, Al, Pb salts of which can be employed.

According to Re. 22,830 an alkali earth metal salt of a sulfur-containing acid of P is employed as addition agent.

U.S.P. 2,414,257. Lubricating Oil. E. A. Evans and J. S. Elliot to C. C. Wakefield and Company, Ltd. (British.)

The extreme-pressure lubricant according to this patent comprises a major proportion of a hydrocarbon mineral oil, to which are added a di- (3-carbomethoxy - 4 - hydroxyphenyl) polysulfide, and a 2-mercaptobenzothiazole or benzothiazole disulfide. The first-named additive brings about the extreme-pressure characteristics of the lubricant, and the second named serves as a corrosion inhibitor. It is used in an amount of less than 0.5 percent which is sufficient to eliminate corrosion of copper and cuprous alloys but insufficient materially to enhance the extreme-pressure properties of the composition. The polysulfide can be employed in amounts of 0.5-2 percent.

U.S.P. 2,415,296. Lubricating Oil B. H. Lincoln and G. D. Byrkit.

The lubricant according to this patent comprises in combination a major proportion of oil of lubricating viscosity and a minor proportion of a salt of a sulfurized relatively-high-molecular-weight unsaturated carboxylic acid. The sulfurization of this acid is carried out by a phosphorous polysulfide containing more than 50 percent S. The oil is stabilized by the addition against oxidation. The calcium salt of sulfurized oleic acid, benzyl-trimethyl ammonium salts of sulfurized corn oil acids, or triphenyl-tin salts of sulfurized soybean oil acids are, for example, employed as additives.

U.S.P. 2,415.353. Rust Preventing Turbine Oil. R. K. Johnston and J. I. Wasson to Standard Oil Development Company.

The mineral lubricating oil according to this patent contains 0.01-1.0 percent of an alkali metal mahogany sulfonate and 0.01-1.0 percent of a polyvalent metal naphthenate, such as zinc naphthenate, dissolved in the oil. The ratio of the quantities of the two additives is adjusted so that the oil blend exhibits substantially no emulsifying tendency in the presence of water when used as a turbine oil.

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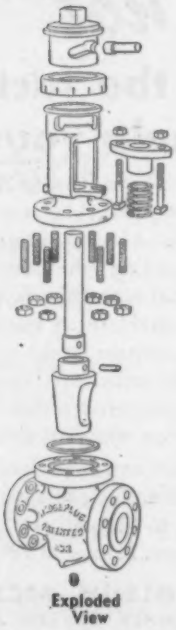
STEEL VALVES

Now in the Popular Price Range

The well-known, tried-and-proved line of Wedgeplug All-Purpose Gear-Operated and Handwheel-Operated Steel Valves is now supplemented by a line of Wrench-Operated Wedgeplug Valves that combine the best features of a Plug Valve with those of a Gate Valve, and yet sell within the popular price range.

Wedgeplug—through the development of a simple, patented plug valve construction—gives you: Large port openings; wedging action; raised seats; and, an easy-to-operate mechanism that lifts, turns, and re-seats the Plug in one operation. The advantages of the "Wedgeplug Principle" have been clearly revealed through actual use under widely-varying and drastic service conditions . . . Write for details of both designs.



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Russian Dictionary

A "Russian-English Technical and Chemical Dictionary," compiled by Ludmilla I. Callaham, is being published by John Wiley & Sons, Inc., New York. In the preface it is stated that "this work is intended chiefly for English-speaking scientists with a fair knowledge of Russian," and since the arrangement of the book is based on the alphabetical order of the Russian words, this would seem to be an accurate description of its utility.

Building Small Skimming Plant in West Texas

R. J. Reischman and associates are erecting a 700-barrel skimming plant near Fort Stockton, Texas, using equipment salvaged from Western Refining Company's abandoned plant at Baldridge, Texas. Crude oil requirements of the plant will be supplied by truck from isolated fields in Pecos and adjoining counties. Reischman currently is operating a 100-barrel skimming plant at the near-by town of Imperial; this unit will be abandoned.

Star Light Refining Company, owner of a 400-barrel skimming plant near Ballinger, Texas, has dismantled the unit and disposed of the usable equipment. Operations were suspended in 1943 because of decline of oil production in the area.

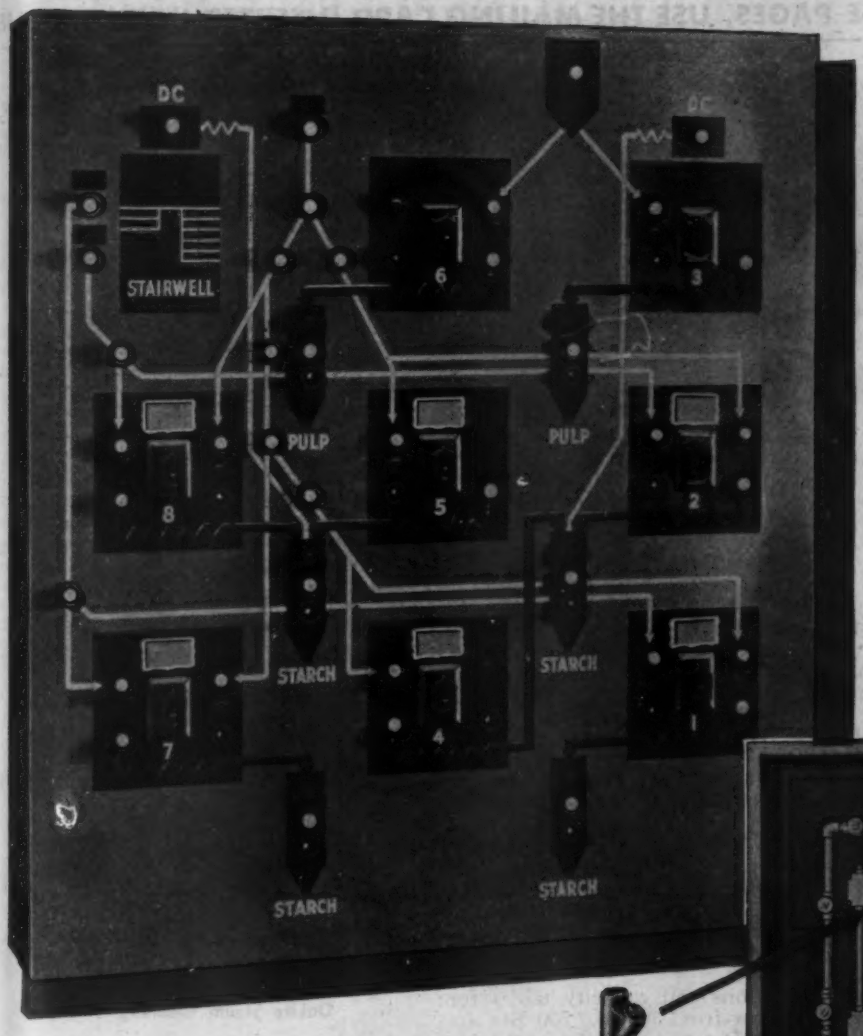
Ink Research Uncovers Another Petroleum Use

Pungent, tarry odors which have limited the use of petroleum drying oils in printing inks are being eliminated by a chemical process developed by I. M. Bernstein, director, and Sam Nelson, research fellow, at the National Printing Ink Research Institute at Lehigh University.

The process, for which a patent is sought, has been discovered in the printing-ink research program concentrating on the development of alternates for, or extenders of, linseed oil at the William H. Chandler chemistry laboratories at Lehigh. Bernstein said several petroleum companies are interested in the process and efforts are being made to start manufacture at an early date.

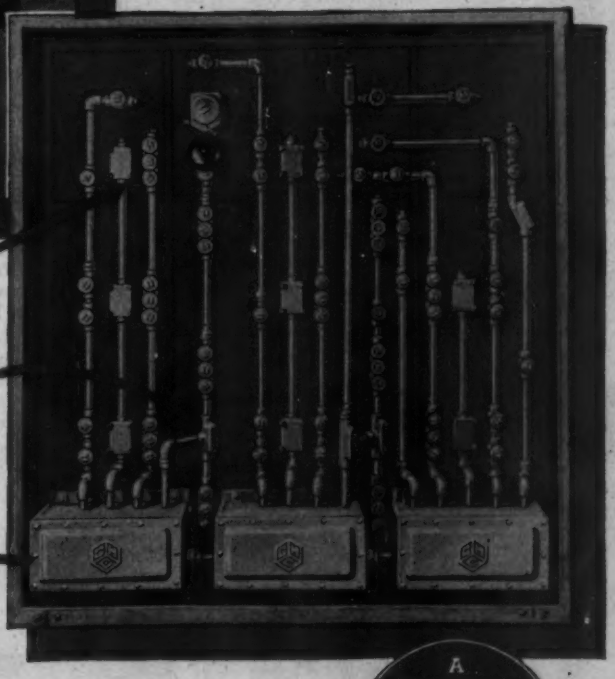
Central flow-line boards for the control of modern industrial processes

are easy to design with CROUSE-HINDS complete line of panel mounting *CONDULET equipment for hazardous locations



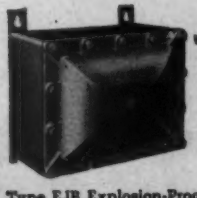
Crouse-Hinds panel mounting pilot lights and switches provide visual indication of process stages and instant control of processing or manufacturing equipment. The illustrations show front and back views of a flow-line control board in a starch plant where combustible dust creates a Class II (National Electrical Code) hazardous location.

*CONDULETS are made only by CROUSE-HINDS



Type EYS Explosion-Proof Sealing Condulet

Type GUAT Explosion-Proof Junction Condulet



Type EJB Explosion-Proof Junction Condulet

The pilot lights and switches are dust-tight and explosion-proof and are designed for both Class I and Class II hazardous locations. A similar control board for use where flammable gases or vapors create a Class I hazardous location would only require substitution of explosion-proof junction Condulets and the addition of explosion-proof sealing Condulets, as indicated. →

Write for additional information.

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1—Transmitters

Taylor Instrument Companies, Rochester 1, New York, announces manufacture of the new Taylor 226R Series Transmitters. The new series provides a practical and economical means of pneumatically transmitting from the point of measurement to a central control panel such process variables as temperature, pressure, rate of flow and liquid level. Simplicity, ease of adjustment, and reliable accuracy are outstanding features claimed for the transmitters, which contain an improved baffle and nozzle assembly to assure dependable performance.

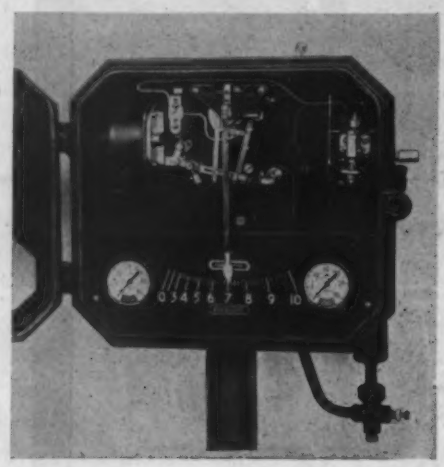
Factory calibration to accuracies within ½ of 1 percent assures straight line relations between pen or pointer movement and output air. Standard range is 3 to 18 psi. Other available ranges are 2 to 14 and 2 to 15 psi.

There are two micrometer screw adjustments—one to establish the range of the instrument, and the other to accomplish zero setting.

The new transmitters (indicating or recording) are available for single or double duty, and as single-duty transmitter-controllers containing any of the five standard forms of Fulscope control.

Bulletin 98156 illustrates and gives detailed information.

CHECK THE CARD AT PAGE 256



Taylor 226R Transmitter

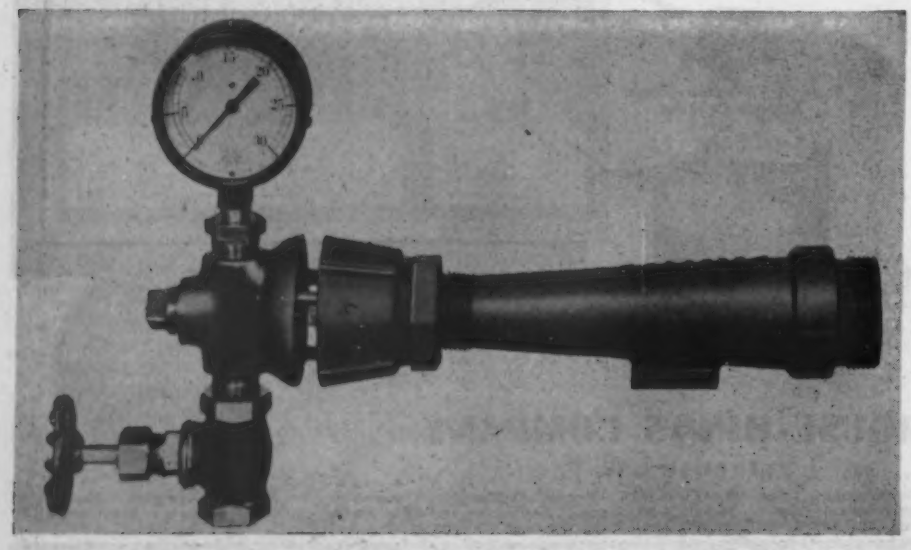
2—High-Pressure Gas Mixer

Bryant Heater Company, Cleveland, Ohio, has announced production of a Venturi mixer for high-pressure gases, incorporating improved mechanical design features. Known as the "Hijector," the unit uses gas under pressures up to 35 pounds to entrain all the air needed for combuestion, and to deliver the mixture to burners at the highest possible pressure.

Overall length of the unit is substantially reduced over conventional designs, by rearrangement of the elements for ease of servicing. By reversing the usual operation of the air shutter and locating it on the body, the orifice spud is accessible for removal without disconnecting any piping. Air-entrainment noise is reduced, while the orifice and air-entrainment areas are readily accessible for inspection or cleaning. The shutter, though backed out of the way, cannot be disassociated from the mixer and become lost. An integral cast mounting boss, with drilled and tapped holes, simplifies mounting of auxiliary devices. The mixer is available in pipe sizes from ¾ to 4 inches.

Specifications and capacity tables for gases ranging from 500 to 2,500 Btu are included in Data Sheet 5B-1, available upon request.

CHECK THE CARD AT PAGE 256



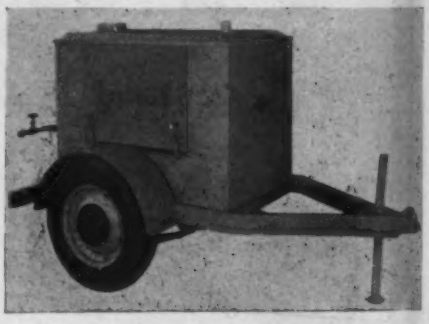
Bryant "Hijector" Gas Mixer

3—Steam-Cleaning Unit

Oakite Products, Inc., 22 Thames Street, New York 6, has announced availability of a new, improved Oakite-Vapor Steam-Cleaning Unit for use in cleaning and paint stripping.

The unit, an enclosed-coil type, downdraft flame steam generator, delivers a hot vaporized spray in either wet or dry state under pressures up to 200 pounds. It generates sufficient steam-cleaning pressure to operate two steam guns simultaneously. The improved model is said to incorporate simplified grouping of gauges and controls; redesigned piping system; plus a compressed-air valve accessory for two-minute anti-freezing of the entire water-steam system. Twelve-gallon capacity of the fuel tank provides ample steam generation for an uninterrupted eight-hour, two-gun cleaning cycle.

After initial throttle setting, unit operation is completely automatic. Downdraft flame in a triple-insulated fire chamber provides rapid, economical steam generation (100 pounds in one minute). Standard dual-chamber steam gun is furnished with the unit; the cleaning solution is aspirated through the gun from a separate solution tank, by-passing the steam coils and eliminating coil-clogging by the cleaning compound. Ample reserve power provides silent, vibrationless operation.



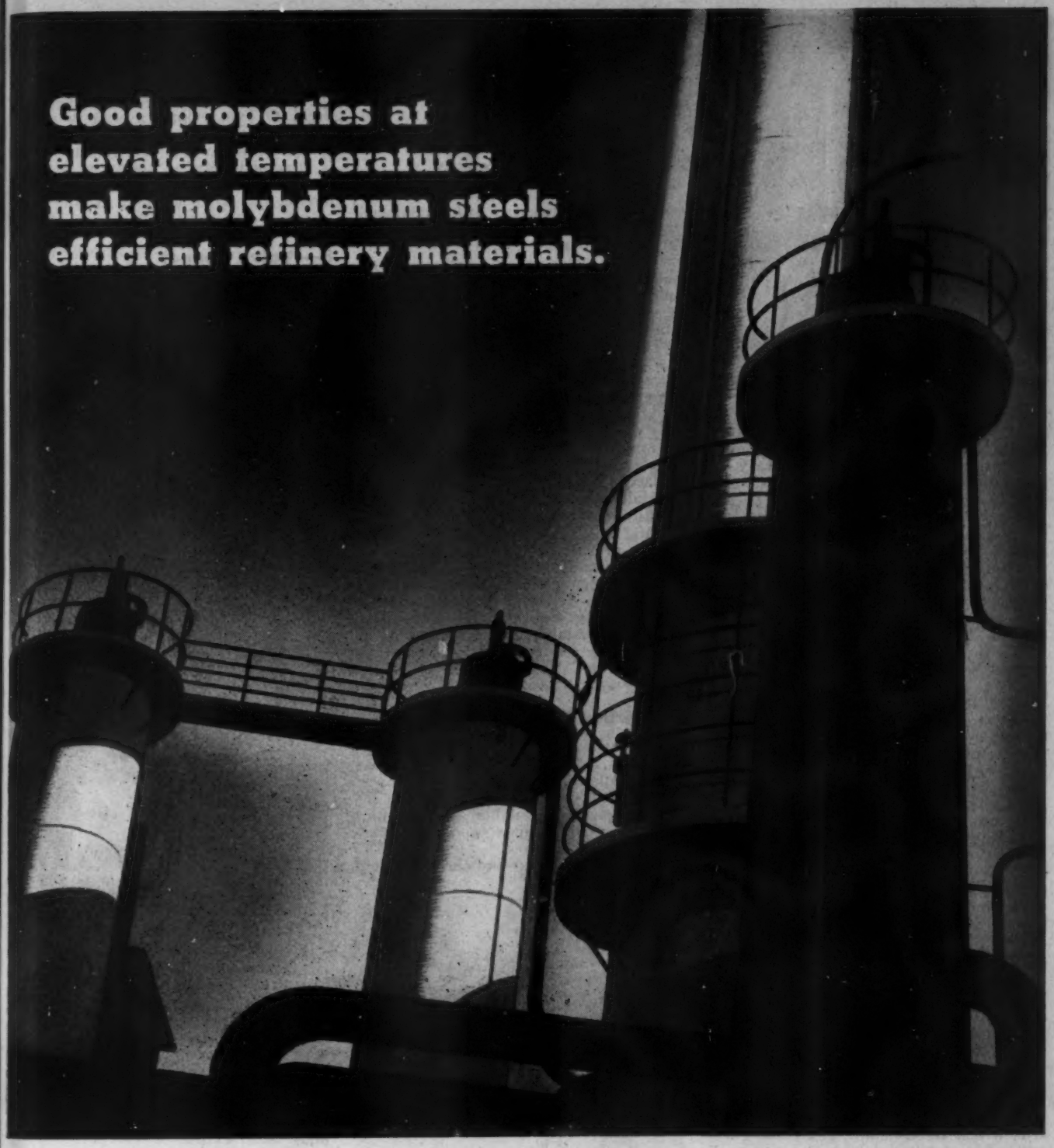
Oakite Steam Cleaning Unit

The unit is available as a stationary model, mounted on shop-wheel chassis for around-the-plant portability; or mounted on a trailer-type chassis. A ¾-horsepower, 60-cycle, 110-220-volt a. c. motor is mounted on the unit. For special applications, alternate motive power includes a self-contained gasoline engine, or special motors.

CHECK THE CARD AT PAGE 256

4—Portable Test Kit

Trutest Laboratories, 20 South 33rd Street, Philadelphia 4, has announced a portable testing unit for analyses of water. The metal cabinet contains apparatus and chemicals needed to make chloride determinations and insure constant carry-over control for high-pressure



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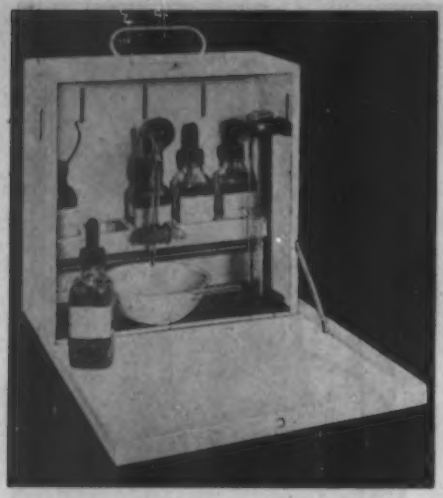


Trutest Water Analysis Kit

boilers. The kit weighs seven pounds.

Determinations by this kit are easy to make, the announcement says. A 100-ml. sample of the water to be tested is obtained by pouring two 50 ml. portions into the casserole. Six drops of phenolphthalein indicator are then added to the sample. If the sample turns pink (indicating alkalinity), 5 percent sulfuric acid is added, drop by drop, until the pink color disappears. Then 10 drops of potassium chromate indicator are added, which turn the sample a bright yellow.

Now N/25 silver nitrate is added, drop by drop, with constant stirring of the sample, from the specially designed automatic micro-burette until a slight orange red color develops throughout the sample. This is the end point. The brick red color resulting from adding more silver nitrate gives a reading past the end point.

Results are read directly from the burette in grains per gallon. For example, if a 100-ml. sample requires 0.4 ml. of N/25 silver nitrate to develop the slight orange red color of the end point, the chloride content of the water is 0.4 GPG.

CHECK THE CARD AT PAGE 256

5—Odor Standards

Cargille Scientific Inc., 118 Liberty Street, New York 6, has announced availability of Crocker-Henderson Odor Standards, offered as a means of specifying the odor of any material or product in terms of a four-digit number. This specification can be checked by different workers at different locations. Odor values are judged by reference to standards which are constant, and results are not influenced appreciably by personal factors. The system was developed in the laboratories of Arthur D. Little, Inc., where it has been in use 20 years.

The system classifies odors according to the intensities of four odor "components:" fragrant, acid, burnt, and caprylic (goat-like). The several thousand combinations of these four components —eight intensities of each—make it possible to assign a specific number to any odor. There are definite ranges of odor numbers for such odors as fishy, rancid, fetid, tarry, oriental, and flowery. Such numerical relations indicate odor values and enable the investigator to perceive, appreciate and describe great numbers of kinds of odors and to handle them intelligently. With a little experience, it was said, only a few minutes are required to evaluate an odor. The standards are permanent. The 32 standards, with extra empty vials, are furnished in a convenient wooden block with transparent plastic cover. Applications suggested include control of odors of petroleum products. Additional information may be had by writing.

CHECK THE CARD AT PAGE 256

6—Glass-Blowers' Lenses

American Optical Company, Southbridge, Massachusetts, has announced availability of AO 6 Curve Super Armorplate Didymium lenses which permit glass workers to look through opaque yellow light emitted by burning sodium and see hidden work areas in glass blowing, fusing and cutting operations.

The lenses absorb more than 90 percent of the yellow sodium flare which conceals details of the job and which is caused by the burning of sodium during glass operations.

By giving the operator a clear view of his work, the lenses help speed up production, reduce spoilage, and prevent headaches caused by eyestrain. They are especially useful in the electronics and glass industries, in laboratory glass blowing . . . on all jobs where sodium flare is prevalent.

In addition to absorbing sodium flare, the AO Didymium lenses have the same high impact resisting qualities as any AO 6 Curve Super Armorplate lens. They are available in orbit shape or 50 mm. round for spectacle or cup-type goggles.

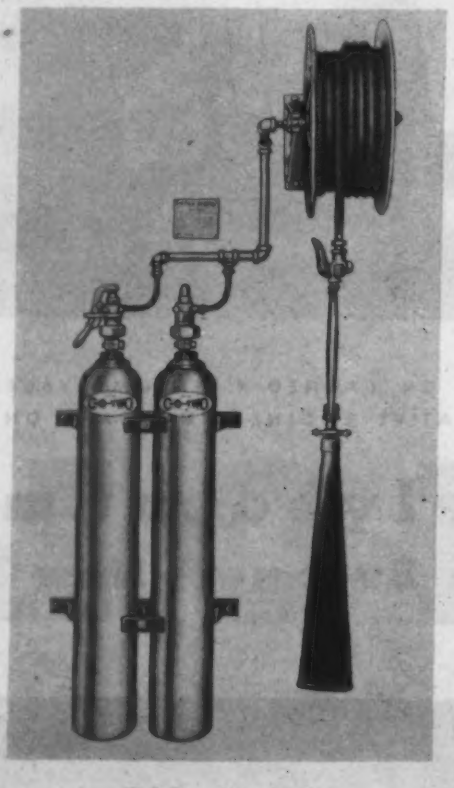
CHECK THE CARD AT PAGE 256

7—Carbon Dioxide Hose Reel

C-O-Two Fire Equipment Company, Box 290, Newark 1, New Jersey, has made available a light-weight hose reel of the shock-proof type for carbon dioxide systems, available in capacities from 50 to 200 pounds or more of carbon dioxide.

This stamped-brass and chrome-plated hose reel may be fitted with ½-inch high-pressure hose of any length up to 125 feet, or any length up to 100 feet of ¾-inch hose. The hose reel and discharge horn may be installed adjacent to the C-O-Two cylinders or at a remote point.

The handle of the fire fighting nozzle is equipped with a "squeez-grip" valve for controlling discharge of the carbon dioxide gas when working around a fire.

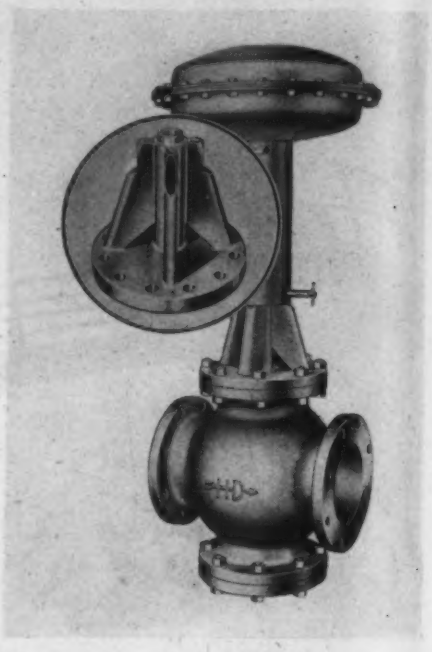


C-O-Two Hose Reel

CHECK THE CARD AT PAGE 256

8—Valve Bonnet

Hammel-Dahl Company, 243 Richmond Street, Providence 3, Rhode Island, announces application of the vertical U type fin radiation bonnet for its diaphragm control valves in sizes 2-inch and larger. The U section permits a draft effect which cools the packing-box section of the valve, and this effect plus increased radiation because of the type of construction reduces packing-box temperatures.



Hammel-Dahl, Radiation-Fin Bonnet

The U fins are fabricated integrally with the forged bonnet and provide strong support for the superstructure.

The manufacturer recommends use of radiation-fin bonnets on control valves when the fluid passing through the valve body is in excess of 450° F. or below freezing.

Additional information may be had by writing.

CHECK THE CARD AT PAGE 256

9—Stainless-Steel Electrodes

Air Reduction Sales Company, 60 East 42nd Street, New York 17, has announced a complete line of stainless-steel electrodes in a full range of grades and diameters.

These electrodes are furnished with heavy extruded-lime type coating for d. c. application. In addition, all but the straight chrome analyses are obtainable with a lime-titania type coating which is useable on a. c. or d. c. Slag produced by either of these coatings is removed easily.

Electrodes with the lime-titania type coating offer many desirable features not

found in the lime type coated electrodes, it was said. For example, satisfactory usability on a. c. eliminates arc blow and results in easier manipulation, more uniform arc action, and better appearance of deposit. This class of electrodes can be recommended for all applications on which the lime type of electrode is used, with the possible exception of highly restrained joints on heavy sections or on steels of high hardenability.

CHECK THE CARD AT PAGE 256

10—Hand-Grip Valve

Airetool Manufacturing Company, 308 South Center Street, Springfield, Ohio, has announced a hand-grip valve to be used with Airetool tube-cleaning motors for cleaning, buffing, grinding and general utility use.

Airetool Hand Grip

The hand grip is equipped with a finger-operated trigger valve that regulates the speed to give easy and simple motor control. This development makes it possible to turn present cleaning motors into useful general utility tools.

Additional information is available by writing.

CHECK THE CARD AT PAGE 256

11—Low-Pressure Test Plugs

Mechanical Products Corporation, 168 North Ogden Avenue, Chicago 7, has announced the "Hydro-Matic" self-sealing low-pressure test plug, a detachable automatic seal for closing openings in vessels requiring internal pressure testing to 100 pounds per square inch. The device is similar to plugs previously offered by this manufacturer for higher pressures. Literature is available giving details.

CHECK THE CARD AT PAGE 256

12—Heat Exchanger

The Duriron Company, Dayton 1, Ohio, has announced production of a corrosion-resisting heat exchanger of new design. It is offered as especially serviceable for heating or cooling small quantities of corrosive solutions over a wide temperature range. The No. 4 size, first of this new line, handles an acid flow of from 4 to 14 gallons per minute, with a heating capacity up to 155,000 Btu per hour, with 75-pound steam and inlet temperature of liquid between 70° and 130° F., and a cooling capacity up to 90,000 Btu per hour, based upon 100°

Now Helicoid offers pressure gages with Monel Bourdon tubes.

This is just one more advantage of Helicoid Gages. Here are some of the others:

- The Helicoid Movement, which eliminates "clock gears."
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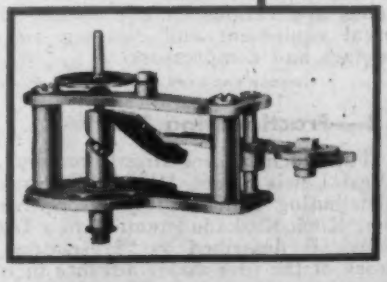
- Fused joints for added strength where it's needed most.
- Silver brazed joints in bronze-tube models for the same reason.
- Bronze tubes of heat-treated, phosphor-bronze with silver brazed joints. (Bronze tubing costs about twice as much as brass, but has better spring characteristics.)
- Socket streamlined for easier cleaning and more equalized stress.
- Weather-proof case to keep out dust and moisture.
- These are just a few of the many reasons why large users of pressure gages are standardizing on Helicoid. Write for our technical catalog which tells the complete story.



The Helicoid Movement is one of many exclusive features of the Helicoid Pressure Gage.

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F. mean temperature differential. Larger units are being developed. The No. 4 unit can be connected in series or parallel.

Construction details, dimensions, installation and operating instructions are contained in Bulletin 1610.

CHECK THE CARD AT PAGE 256

13—Alkalinity Test

Trutest Laboratories, Inc., 20 South 33rd Street, Philadelphia 4, has issued a bulletin describing a new indicator for fast determination of alkalinity of water.

CHECK THE CARD AT PAGE 256

14—Oxygen Breathing Apparatus

Mine Safety Appliances Company, Pittsburgh 8, has available Bulletin B-14, descriptive of the MSA Chemox Oxygen Breathing Apparatus. It is a self-contained breathing apparatus with a replacable canister which generates oxygen for a minimum of 45 minutes.

CHECK THE CARD AT PAGE 256

15—Seamless Welding Fittings

Ladish Company, Cudahy, Wisconsin, has issued a 10-page folder, giving dimensions and approximate weights on a recently introduced line of controlled-quality seamless welding pipe fittings. Already stocked by distributors in principal centers, the new line includes 90- and 45-degree elbows, 180-degree return bends, straight and reducing tees, concentric and eccentric reducers, caps, lap joint stub ends, saddles, shaped nipples, crosses and tees in carbon steel; sizes up to 30 inches.

CHECK THE CARD AT PAGE 256

16—Packing

Raybestos-Manhattan, Inc., Manheim, Pennsylvania, has issued a folder, Form A909, giving data on its "Vee-Flex" automatic packing rings for hydraulic cylinders, triplex pumps and heavy-duty valve stems.

CHECK THE CARD AT PAGE 256

17—Air Filters

American Air Filter Company, 215 Central Avenue, Louisville 8, Kentucky, is distributing a new 28-page illustrated booklet which discusses various types of industrial dust problems and typical applications of the company's air filters to such problems. Included is a chart of size and characteristics of air-borne solids. Sections dealing with miscellaneous applications include cooling electrical equipment and cleaning air for engines and compressors.

CHECK THE CARD AT PAGE 256

18—Fractionating Tray

The Koch Engineering Company, 335 West Lewis Street, Wichita, Kansas, is distributing Bulletin 101, descriptive of the "Koch Kaskade Fractionation Tray," which is described as "presenting the story of the first major advance in fractionating technique in 30 years."

CHECK THE CARD AT PAGE 256

19—Liquid Conditioning

Liquid Conditioning Corporation, 114 East Price Street, Linden, New Jersey, has available a condensed catalog which gives information on methods and equipment for treatment of water, purification of process liquids, and recovery of valu-

BLAW-KNOX
Aquatomic
FOG NOZZLES DO NOT FAIL!

Wide clear passages through the heart of this dependable sprinkler head make it virtually non-clogging—always reliable.

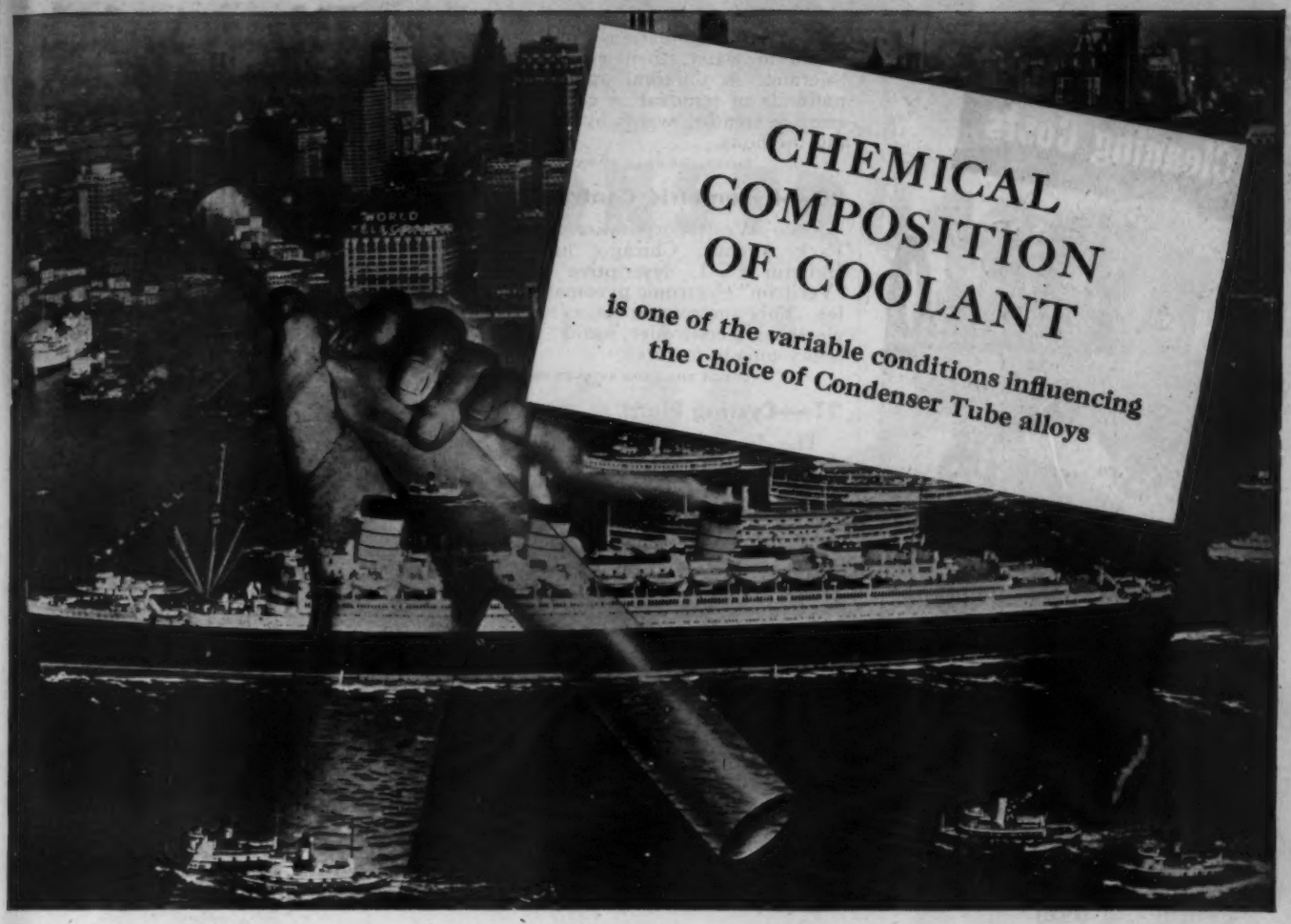
It fights fire with the water available—maintains its spray pattern under low pressures in emergency.

Fine but forceful spray gives maximum cooling and quenching effect and drives to the base of the fire in spite of drafts and turbulence.

Also—
Blaw-Knox offers Standard Wet and Dry Pipe Automatic Sprinkler Systems, as well as Thermostatically Controlled Pre-Action and Deluge Systems.

Write for details.

BLAW-KNOX
SPRINKLER DIVISION
832 Beaver Avenue, N.S.,
Pittsburgh 12, Penna.



This coolant may be distilled and relatively chemically inert fresh water, as in the case of air ejector inter- and after-coolers in certain Marine and stationary power plants, or relatively clean sea water containing chlorides and sulfates of sodium, potassium, magnesium and calcium with minor quantities of other salts, in a condenser aboard a ship under way. It may be brackish water containing not only the chemicals found in natural fresh and sea waters, but often the more chemically active waste products of industrial plants; or, in an oil refinery heat exchanger, it may be crude oil absorbing the heat of vaporization of one or more of the fractionated constituents.

Whatever the chemical composition of this medium may be, it is not the sole determinant of tube life in an actual installation, but must be considered in combination with some or all of such other factors as:

Velocity and turbulence of coolant . . . Abrasives suspended in coolant . . . Dissolved or entrained air or gases . . . Temperatures inside and outside of tubes . . . Marine growths or other deposits . . . Development of protective film . . . Media surrounding tubes.

One of the principal objects of Condenser Tube Technical Research has been the development of alloys designed to give longest life and most trouble-free service. Today's copper-base condenser tube materials, with an occasional resort to an alloy not usually considered in this connection, cover a wide range of uses, not only in the Marine and stationary power industries, but in the chemical and petro-chemical fields as well.

The conditions surrounding any given Condenser Tube installation are many and varied, so that each installation must be treated as an individual problem.

Scovill has made, or is making, extensive studies of Condenser Tube service conditions for every known application, and offers the facilities of its Technical Service Department in the selection of suitable Condenser Tube alloys to meet specific service conditions in an existing or proposed installation.

THREE SCOVILL SERVICES

Scovill's *Service in Men* offers experienced engineering to help solve your tube selection and installation problems. *Service in Metals* develops and produces Scovill tube alloys that conform to your requirements. *Service in Manuals* consists of literature which gives you latest, authentic information on condenser and heat exchanger tubes. A Condenser Tube Booklet is available. Scovill Manufacturing Co., 15 Mill St., Waterbury 91, Conn. *Export Department:* 405 Lexington Ave., New York 17, N. Y.

SCOVILL

SCOVILL

HEAT EXCHANGER TUBES

One Product • Three Services

Service in Manuals . Service in Metals . Service in Men

with the New Oakite-Vapor Steam Cleaner

Outstanding features of this trailer-mounted steam generator help you save time and money in cleaning drilling rigs, slush pumps, derrick gears, tanks, sheds, tractors:

100 lbs. pressure in 1 minute (rated capacity 200 p.s.i.)

8-hr. non-stop TWO-gun cleaning or paint-stripping without refueling

Fully automatic pressure and temperature safety controls

Cleaning compound by-passes heater coils . . . no danger of clogging

Easy, 2-minute anti-freezing of water-steam system

Quiet, no-creep, automatic operation

TRAILER-MOUNTED UNIT: Rugged, spring-suspended trailer chassis has pneumatic tires, sturdy steel tow frame. Unit is furnished with ¾ hp, 110-220 v. ac motor or, if power supply unavailable, with self-contained gasoline engine. Immediate deliveries. Free Folder illustrates both stationary and portable models.

OAKITE PRODUCTS, INC.
30 Thames Street, NEW YORK 6, N. Y.
Technical Representatives in Principal Cities of U. S. & Canada

OAKITE

Specialized Industrial Cleaning
MATERIALS • METHODS • SERVICE

able substances from waste liquids. Included are tabulations of impurities found in water, their effects, limits of tolerance in different applications, and methods of removal. A chart shows results of treating water by six conditioning methods.

CHECK THE CARD AT PAGE 256

20—Pyrometric Controller

Taco West Corporation, 2620 South Park Avenue, Chicago, has available Bulletin PC-1, descriptive of its new "Veritron" electronic pyrometric controller. This instrument is a two-position electronic controller, said to include many unique features.

CHECK THE CARD AT PAGE 256

21—Cycling Plant

The Fluor Corporation, 2500 South Atlantic Boulevard, Los Angeles 22, has available a 32-page brochure on the South Coles Levee Cycling Plant, which it designed and built. In addition to many photographs accompanying the descriptive text, there is a four-color, streamlined flow sheet which graphically depicts the various stages required in processing the well gas and condensate.

CHECK THE CARD AT PAGE 256

22—Water Pumps

Byron Jackson Company, Box 2017 Terminal Annex, Los Angeles 54, has issued Bulletin 46-6000, "The Hydropress Pump," for those who have big-pressure, low-volume pumping jobs, requiring maximum efficiency. Pump sizes covered are 10- 12- and 15-inch, with capacities from 20 to 300 gallons per minute, and head ranges 55 to 250 feet per stage (to pressures of 5,000 pounds per square inch).

CHECK THE CARD AT PAGE 256

23—Water Softeners

Cochrane Corporation, 17th and Allegheny Avenue, Philadelphia 32, has issued Publication 3000—"Cochrane Hot Process Softeners," a 66-page treatise on feed-water treatment. In addition to discussions of variations in the treatments that may be applied and designs applied, it contains tables and data of interest to chemists.

CHECK THE CARD AT PAGE 256

24—Pyrometers

Leeds & Northrup Company, 4934 Stenton Avenue, Philadelphia 44, is distributing Catalog ND46(1), "Speedomax Type G Pyrometers," a 16-page illustrated booklet designed to provide information about a pyrometer which supplements the company's "Micromax" line of instruments for applications which require split-second response to temperature changes, or unsually rapid concentration on a single chart of data from many points.

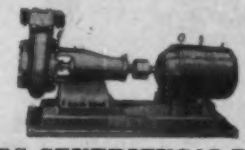
CHECK THE CARD AT PAGE 256

25—Creep Stress Data

The Babcock & Wilcox Tube Company, 85 Liberty Street, New York 6, has available a new data card (104-A) which shows, graphically, creep stress data for B & W Croloy 2¼ and approximate comparison of creep strength data for the various heat-resistant B & W Croloy steels. The company points out that this card is for general or comparative purposes only, and is not designed for use where precise figures are required. Other cards and bulletins showing exact figures for safe design are pub-

Plan with Peerless!

Peerless Centrifugal Pumps
(formerly Dayton-Dowd)
Meet extensive, continuous-duty pumping requirements in all Industries and Municipalities desiring advanced pump design, reduced operating cost and high pump efficiency.



Type DS CENTRIFUGAL PUMPS
For Chemical, Oil, Food Processing, etc.
Capacities: Up to 600 G. P. M. Heads to 230 ft.

Type XT TURBINE (Vane) PUMPS
For Butane-Propane, General and Process Work.
Capacities: Up to 150 G. P. M. For high heads.

Type A CENTRIFUGAL PUMPS
For General Services-Maximum efficiencies.
Capacities: Up to 60,000 G.P.M. Sizes: 2" to 42".

Type B BOILER FEED PUMPS
For High Pressures in Refinery, Pipe Line and Boiler Feed Services. Pressures up to 7001bs.

Type AF FIRE PUMPS
(formerly Dayton-Dowd)
Underwriters Approved. Capacities 500 to 2000 G. P. M. One of a Variety of Peerless Fire Pumps.

PEERLESS Pump Division manufactures an extensive line of high quality centrifugal pumps for usual or unusual pumping applications. The Peerless staff of highly trained engineers or field representatives will gladly assist you in prescribing the proper pump for your specific application. If you are considering a pump installation demanding long range, trouble-free service, then Plan With Peerless—It will pay!

PEERLESS PUMP DIVISION
FOOD MACHINERY CORPORATION
Factories: 301 W. Ave. 26, Los Angeles 31, California: Quincy, Ill.; Canton 6, Ohio—District Offices: Canton 2, Ohio; Ardmore, Pa.; Decatur, Georgia; Dallas, Texas
Distributors in Principal Cities

TOP STRENGTH

IN TOP CONSTRUCTION

OHIO TYPE 1640

★ **The top construction of Ohio Type 1640 Return Bend is the strongest type in use as shown by its higher pressure temperature rating. And the entire fitting has been made even stronger by applying the principle of directional solidification in the casting operation. This stronger casting is free of center line shrinkage or weakness found in ordinary designs.**

The combination of these two features means longer life for your return bends.

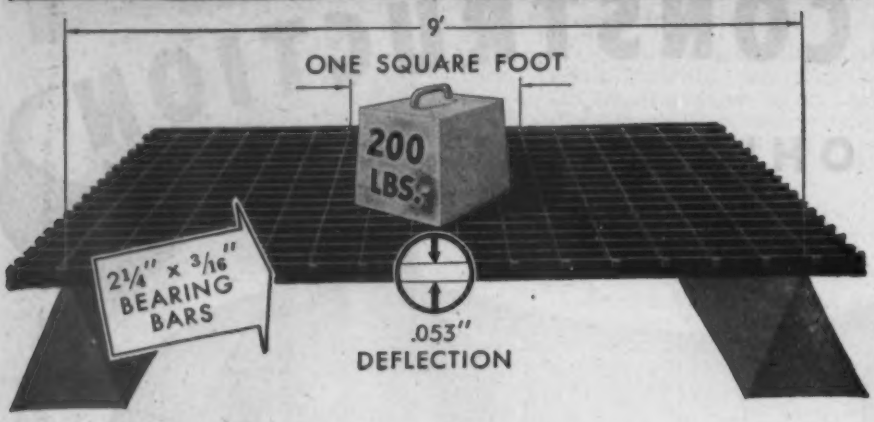
WRITE FOR THE FACTS

THE OHIO STEEL FOUNDRY COMPANY

SPRINGFIELD, OHIO

PLANTS IN LIMA AND SPRINGFIELD, OHIO

This is TRI-LOK OPEN STEEL FLOORING



The locked in strength of TRI-LOK enables it to stand up under heavy loads—even on long spans. No rivets, bolts or welds are used in its construction, thus, the possibility of loose joints is eliminated. *Write for Bulletin 1140.*

DRAVO CORPORATION
NATIONAL DEPARTMENT
300 Penn Avenue, Pittsburgh 22, Pa.
(Distributor for THE TRI-LOK COMPANY)

DRAVO

30 Years STEAM JET VACUUM EXPERIENCE

The Croll-Reynolds Company was originally founded in 1917 by engineers who had already accumulated specialized experience in the design and manufacture of steam jet vacuum equipment. Since then this organization, still under the active leadership of the original founders, has specialized on this particular item, to a very exceptional degree. The applications of steam jet vacuum equipment have increased tremendously in this time. Great progress has also been made in improving the simplicity and efficiency of the equipment. Even greater in importance is the wider range of vacuum which can be produced. Almost every year finds commercial equipment producing vacuum higher than was thought possible outside of laboratories.

While the steam jet vacuum equipment is not suitable for the extremely low pressures obtained with diffusion pumps, it has been developed to the point where it overlaps the applications of this equipment, particularly at pressures above 100 microns. From this pressure through all the intermediate range of vacuum up to atmospheric pressure, Croll-Reynolds EVACTORS are handling a great variety of industrial requirements, and frequently helping to pioneer new industrial processes. Inquiries are solicited on this equipment, also on all types of steam condensers, and on the Croll-Reynolds CHILL-VACTOR unit for flash cooling of water and aqueous solutions.

CROLL-REYNOLDS CO.
17 JOHN STREET, NEW YORK 7, N. Y.
CHILL VACTORS STEAM JET EVACTORS CONDENSING EQUIPMENT

CROLL-REYNOLDS

lished by the company and are kept up to date through frequent revisions. The new card does not replace, but supplements such information. Technical data cards, bulletins and other literature covering problems arising in connection with use of seamless or welded tubing are available upon request to sales offices of the company.

CHECK THE CARD AT PAGE 256

26—Color

Pittsburgh Plate Glass Company, 632 Duquesne Way, Pittsburgh 22, has published a colorful 32-page booklet which shows how scientific use of the energy in color is employed in plants to increase over-all efficiency. Sections are devoted to use of color on machinery and equipment, walls, ceilings, floor, aisles and mobile equipment with emphasis on color for safety. A special section contains material specifications for industrial painting.

CHECK THE CARD AT PAGE 256

27—Water Degasification

Cochrane Corporation, Philadelphia 32, has available Publication No. 4076—The Degasification of Water by Cochrane Equipment.

CHECK THE CARD AT PAGE 256

28—Temperature Regulators and Controllers

Leslie Company, Lyndhurst, New Jersey, has issued Bulletin No. 464—"Engineering, Operating and Maintenance Data on Temperature Regulators and Controllers."

CHECK THE CARD AT PAGE 256

29—Solderless Wiring

Aircraft-Marine Products Inc., 1523 North Fourth Street, Harrisburg, Pennsylvania, has available a selection data book on its line of solderless wiring devices for installing terminals in a variety of wiring applications.

CHECK THE CARD AT PAGE 256

30—Pressure-Reducing Valves

A. W. Cash Valve Manufacturing Corporation, 666 East Wabash Avenue, Decatur, Illinois, has issued Bulletin No. 199, descriptive of its line of small pressure-reducing valves.

CHECK THE CARD AT PAGE 256

31—Pipe

"Pipe in American Life," a 48-page illustrated booklet published by the Committee on Steel Pipe Research of American Iron and Steel Institute, presents the historical background and modern uses of metal pipe, with emphasis on the use of steel pipe.

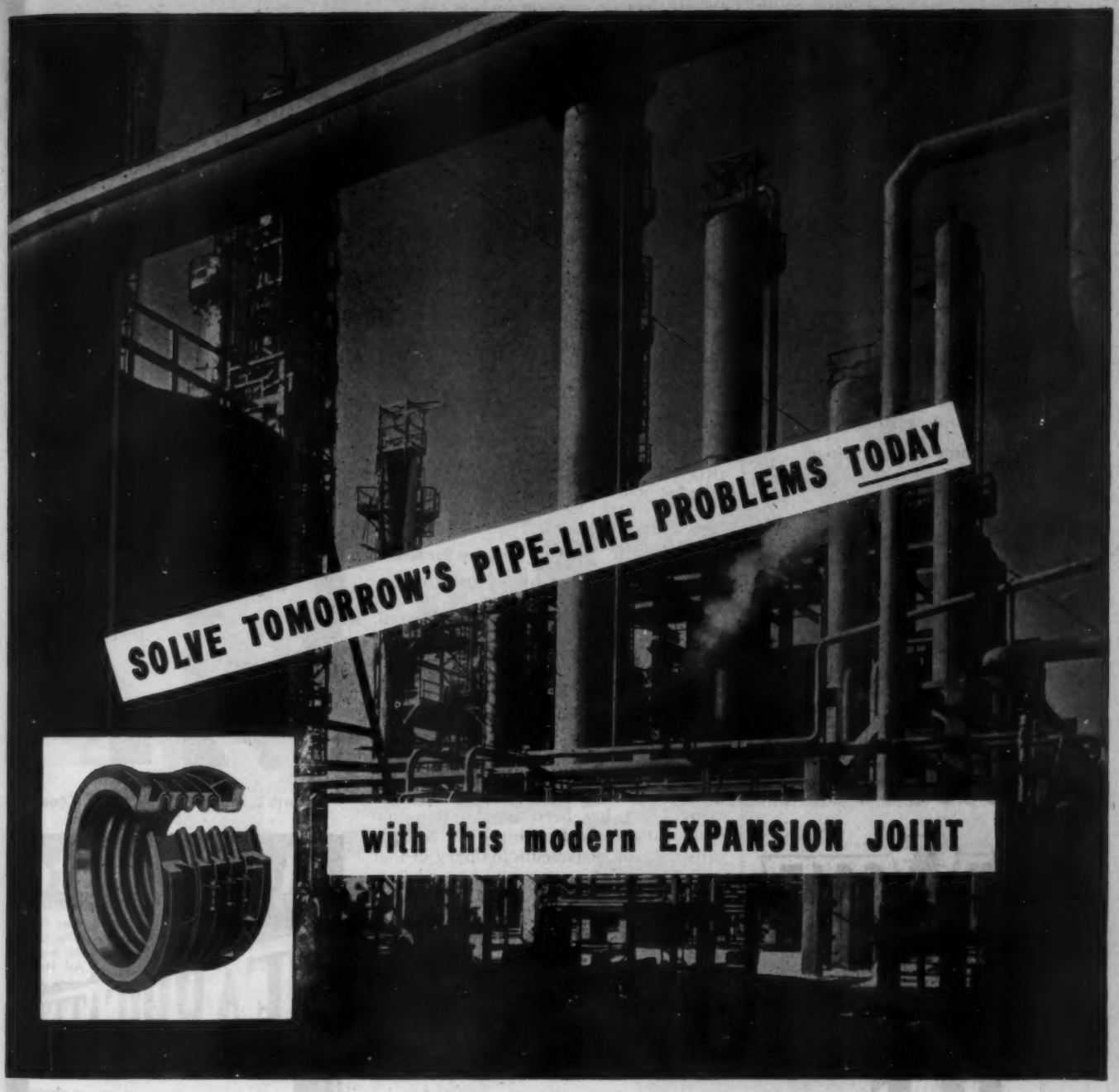
Much of the material has not previously been available as general information. Copies are available, American Iron and Steel Institute, 350 Fifth Avenue, New York 1.

CHECK THE CARD AT PAGE 256

32—Tank Roof

"Vapor Saving Roofs" is the title of a new eight-page bulletin recently made available by the Graver Tank & Manufacturing Company. This company's expansion roof and improved floating roof of double-deck construction are described along with information on the conservation of petroleum products in storage.

CHECK THE CARD AT PAGE 256



• *E. B. Badger & Sons Co. is the original and sole manufacturer of* **BADGER** *Expansion Joints.*

ONCE you install dependable, long-lasting Badger Packless Corrugated Expansion Joints, your pipe expansion problems are solved.

The flexing member of every one of these joints is made from a single piece of tubing; hence *no packing is required.* This eliminates costly and troublesome maintenance—assures long service without attention.

Other important advantages of the Badger Expansion Joint are its *simplicity, compactness* and *ease of installation.*

Badger Packless Expansion Joints for the Petroleum industry are made of stainless steel and other alloys to withstand high temperatures and resist corrosion.

A special heat treatment, after forming, further assures their resistance to corrosion, thereby lengthening their life of service.

For more information about these "life-time" expansion joints, write for Bulletin No. 100.

Badger

E. B. BADGER & SONS CO., 75 Pitts Street, Boston 14, Mass.
AGENTS IN PRINCIPAL CITIES

PACKLESS CORRUGATED EXPANSION JOINTS

E. B. BADGER & SONS COMPANY
WEATHER VANE ON FANEUIL HALL
TRADE MARK
ESTABLISHED 1841

ENGINEERS

E. B. Badger and Sons Company has openings for several engineers with experience in the design and construction of chemical plants and petroleum refineries.

Write fully of your education and experience to:

Personnel Director

E. B. BADGER & SONS CO.

75 PITTS ST., BOSTON, MASS.

All replies will be treated in strict confidence.

PENN SALT

Anhydrous

HF

Effective and practical alkylation catalyst for production of high octane motor fuels.

•

Also finding use as condensation reagent. Of increasing interest in polymerization, isomerization and acylation reactions.

•

Write for helpful booklet, "How to Handle Anhydrous Hydrofluoric Acid Safely."

•

PENNSYLVANIA SALT MANUFACTURING COMPANY

1000 WIDENER BUILDING, PHILADELPHIA 7, PA.

New York • Chicago • St. Louis • Pittsburgh
Cincinnati • Wyandotte • Tacoma • Portland, Ore

About EQUIPMENT—SERVICE Suppliers

Cooper-Bessemer Corporation Advances Horace Sanders

The Cooper-Bessemer Corporation has announced promotion of Horace Sanders to assistant Southwestern district manager.

Horace Sanders

Sanders has been with the organization 20 years, starting as stenographer and warehouseman for Bessemer Gas Engine Company at Shreveport. In 1931, after the consolidation which formed The Cooper-Bessemer Corporation, he was transferred to Dallas, and since 1933 he has devoted his efforts to sales and administrative activities. In his new capacity he is assistant to A. A. Burrell, Southwestern district manager for the company at Dallas.

Reading-Pratt & Cady Sales Setup in Southwest Changed

J. A. Bynum, who has been district sales manager for Reading-Pratt & Cady Division of American Chain & Cable Company, Inc., in the Southwest for many years, has been appointed special representative on Reading-Pratt & Cady valves for the petroleum industry in that area.

J. J. Reed has been appointed district sales manager to succeed Bynum. For most of the past five years Reed has been located at the factory at Reading, Pennsylvania. Prior to that time he was engaged in sales work in the Southwest. Both Bynum and Reed will make headquarters at the Houston office of the company, 630 McFarland Street.

National Tube Company Sales Personnel Promotions Announced

Forrest A. Marsh, division sales manager for National Tube Company at Houston, has been appointed assistant to the vice president in charge of sales for the company, headquarters at Pittsburgh. He succeeds James B. Graham, who is retiring. Earle E. Smith has been moved from Tulsa to succeed Marsh at Houston; G. F. Coons, formerly at New Orleans has become sales manager at Tulsa, and Thomas W. Thorne promoted to fill the New Orleans post.

F. A. Marsh

Marsh, who attended Purdue University, joined National Tube Company in 1924 and worked in the mills five years until he was appointed a field engineer. He became division sales manager at Houston in 1936 and has been manager of the office there since 1944.

Smith began with the company as a machinist, following graduation from Carnegie Institute of Technology with a degree in mechanical engineering. He held several engineering and operating positions at the plant until 1929, when he was made a field engineer in Texas. Three years later he became a sales agent at Tulsa, and later was transferred to Pittsburgh as director of service. He was appointed manager of sales at Tulsa four years ago.

Coons has been manager of the New Orleans sales office since 1942. He was graduated from Wyoming State Univer-

Earle E. Smith

G. F. Coons

Multi-Metal

Wire Cloth
Filter Cloth
All Meshes
All Metals

FABRICATED WIRE CLOTH

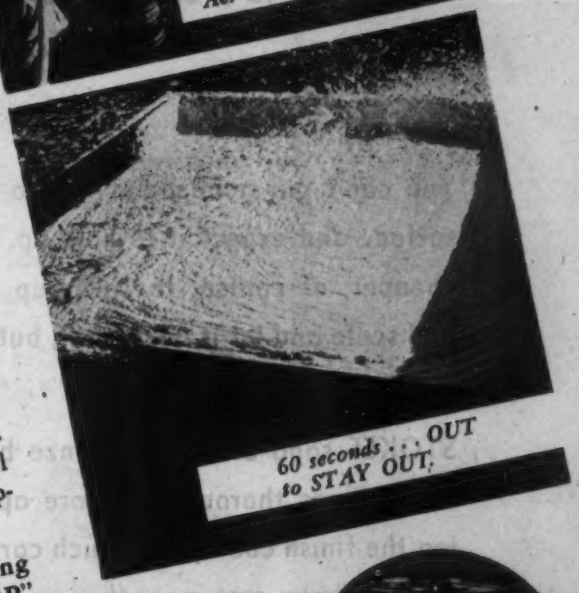
For more than 34 years Multi-Metal has designed and fabricated an infinite variety of filter units which involve the use of wire cloth. Multi-Metal stresses its technical facility. In addition to our fabrication plant, we carry a wide variety of wire cloth which is available by the roll and cut piece. Write for our new catalog and free wire cloth samples.

Multi-Metal
WIRE CLOTH COMPANY, INC.
1350 Garrison Ave., New York 59, N. Y.

60 seconds to go... then OUT by Aer-O-Foam!

A gasoline tank blazing away...

30 seconds... after Aer-O-Foam application.



60 seconds... OUT to STAY OUT.

THAT'S the kind of fire-fighting effectiveness that has made NATIONAL Foam the standard method for fighting oil fires in the petroleum industry for years.

NATIONAL Aer-O-Foam, a *non-corrosive* mechanical foam, quickly forms a thick, tough, lasting blanket that smothers a fire . . . insulating threatened areas and preventing flashbacks. It flows quickly and clings fast even to vertical surfaces . . . will form a fog-foam if required. Aer-O-Foam will not harm machinery . . . can be cleaned off equipment, walls and floors.

For a simple, dependable means of applying Aer-O-Foam, NATIONAL has developed its "RP" Type Nozzles, easily handled by one man. These nozzles, provided with pick-up tubes, deliver a spot pattern stream, with no weeping.

Let us know your problem . . . we'll offer specific protective measures.

NATIONAL FOAM SYSTEM, INC.

Specializing in Foam Fire Protection

Packard Building, Philadelphia 2, Pa.

sity, and was employed by several oil companies and The National Supply Company before joining National Tube Company as a field engineer in 1930.

Thorne was graduated from North Carolina State College with a degree in mechanical engineering. He started with the company as a sales trainee, and has held position as a salesman at Atlanta, New Orleans and Houston.

Wheelco Instruments Company Makes British Tie-Up

Wheelco Instruments Company, Chicago, has consummated an arrangement whereby its instruments will be manufactured and marketed in the United Kingdom by Ether, Ltd., Birmingham, England.

Neilsen & Fryer Representing Hammel-Dahl in Chicago Area

Hammel-Dahl Company has announced appointment of Neilsen & Fryer, Inc., 152 East Superior Street, Chicago 11, as its dealer representative in the Chicago area.

Neilsen & Fryer, Inc., organized in 1945 as specialists in sales and service of instruments and automatic control equipment, includes D. M. Neilsen and J. C. Fryer.

Neilsen has an electrical engineering degree from Rensselaer Polytechnic Institute, and for eight years previous to his present association was with the sales department of The Foxboro Company. He has been a contributor to the literature on automatic controls.

Fryer obtained a degree in electrical engineering and engineering mathematics from University of Michigan in 1933. From 1934 to 1936 he was with Schweitzer & Conrad, and later was sales and service engineer for The Foxboro Company at Chicago.

J. C. Fryer

D. M. Neilsen

THE PART YOU CAN'T SEE

Is what makes a

GOOD PAINT JOB

●

You can't put good paint over a bad surface and expect it to hold up. It's cheaper, of course, to cover up rust and scale and let it go at that, but it's mighty expensive in the end.

STOKES sand blasts, or bronze buffs all surfaces thoroughly before applying the finish coat. While such careful methods may cost more they save you money in the long run.

Inquiries invited.

R. A. STOKES INC.

635 NIXON BLDG. CORPUS CHRISTI, TEXAS

Phones 2-2101 and 5316

Spence Elected President Mechanical Packing Association

Elmer L. Spence was elected president of the Mechanical Packing Association at its recent annual meeting in New York.

Spence, manager of the Packing Department of United States Rubber Company, was formerly a director and chairman of the association's standards and customs committee.

A. W. Swartz, Sr., of Linear, Inc., Philadelphia, was elected vice president, and F. H. Luhrs, New York, secretary-treasurer. Directors elected are C. A. G. Pease, Endura Manufacturing Corporation, Charles E. Cunningham, Flexitallic Gasket Company, and A. R. Byrnes, Union Asbestos & Rubber Company.

Fairbanks, Morse Appoints New Los Angeles Manager

John A. Cuneo has been appointed manager of the Los Angeles branch of Fairbanks, Morse & Company. He succeeds Harry W. Brown, who has retired.

Cuneo joined the company in 1929 as a field engineer in the export division, traveling the West Indies and Latin America. Later he was manager at Havana, following which he was assigned to New York, Chicago and St. Louis before being made assistant manager of the export division in New York. During the past two years he was a member of the organizing staff of the company's subsidiary in Mexico.

Jones & Laughlin Supply Makes New Sales Assignments

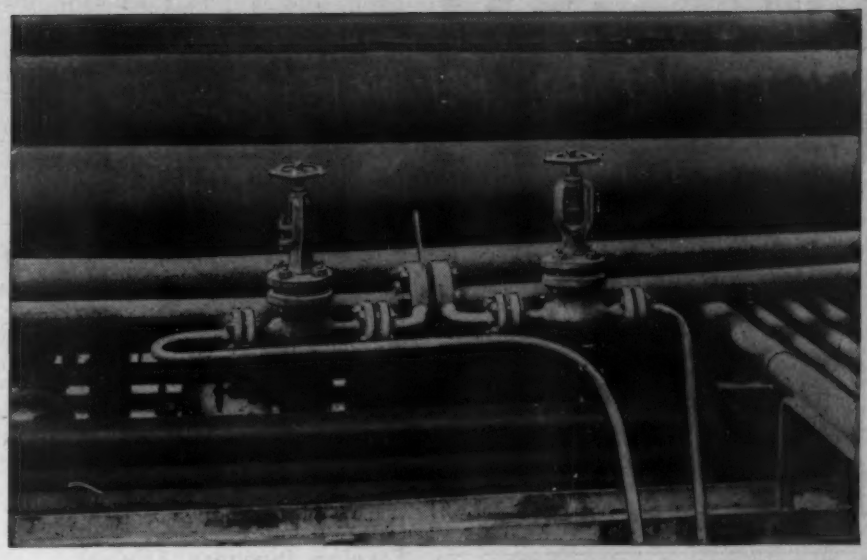
H. L. Wilkins and A. G. Bastian have been named assistant sales managers of Jones & Laughlin Supply Company. Wil-

H. L. Wilkins

A. G. Bastian

3 useful metals

for valves and fittings in HF alkylation units



BOOSTING VALVE LIFE. Globe valves with "K" Monel stems, discs, disc nuts and seats increase valve life and reduce maintenance problems. "K" Monel is one of three INCO Nickel Alloys especially suited for use with hydrofluoric acid.

At least three commercial nonferrous metals provide adequate resistance to hydrofluoric acid.

They are Monel*, "K"* Monel and "S"* Monel.

Producers of hydrofluoric acid and operators of HF alkylation units find them useful against this destructive corrosive's dilute and anhydrous forms.

Monel

Like all high-Nickel alloys, Monel is tough and erosion resistant. It has a tensile strength of 75,000-85,000 psi, yet can be readily fabricated and welded. At temperatures up to 250° F., where oxygen is absent and sludge is not allowed to accumulate, it resists attack by HF in all concentrations.

In rerun systems where quantities of water are present and ordinary valves corrode badly, Monel's corrosion rate is noticeably lower than that of other commercial nonferrous metals.

"K" Monel

All the properties of Monel—and more besides — are present in "K" Monel. Through heat treating, it gains *extra* strength and hardness without sacrificing any of its corrosion resistance.

"S" Monel

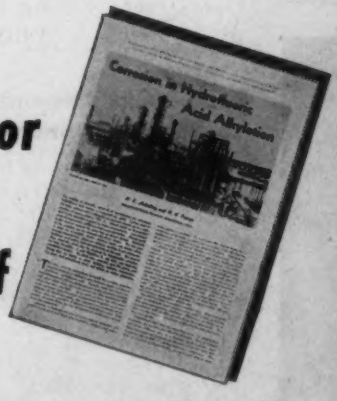
For certain purposes, the superior hardness and non-galling properties of cast "S" Monel are desirable. This alloy also displays excellent resistance to HF corrosion.

Let INCO help you

Investigate the applications of Monel, "K" Monel and "S" Monel for HF service. And for help with your problems, call on INCO's Technical Staff. A note to them at the address below will make their accumulated experience available to you without fee or obligation.

THE INTERNATIONAL NICKEL COMPANY, INC.
67 Wall Street, New York 5, N. Y.

Send for FREE copy of



This authoritative article contains photos, tables and metallurgical information of permanent value to operators of HF alkylation units. It's free, but supply is limited.

DON'T DELAY—WRITE TODAY

CLOSE-UP of one of the forged steel globe valves with "K" Monel trim fabricated by HENRY VOGT MACHINE CO., Louisville, Ky., for HF service in refinery alkylation units.

NICKEL (INCO TRADE MARK) ALLOYS MONEL* • "K"* MONEL • "S"* MONEL • "R"* MONEL • "KR"* MONEL • INCONEL* • NICKEL • "L"* NICKEL • "Z"* NICKEL

*Reg. U.S. Pat. Off.

QUICK TURBINE REPAIRS

If you need dependable Turbine repairs *in a hurry*, call us. We are completely equipped to repair and dynamically balance turbine rotors and any High Speed rotating elements in our modern shops.

25 Years Successful Experience

GULF ENGINEERING CO., INC.
"Service Not Promises"
1941 INGEBORG ST. HOUSTON, TEXAS
916 S. PETERS STREET NEW ORLEANS, LA.

RUST-OLEUM
POSITIVELY PREVENTS RUST

Longer life for tanks, buildings, structural steel, machinery, pipes, stairways, loading racks, fences and every metal surface.

(See page 968—Refinery Catalog)

RUST-OLEUM
CORPORATION
EVANSTON, ILLINOIS
TEXAS BRANCH
1935 Commerce St., Dallas, Texas

McNamar
Steel Plate
Fabricators and
Contractors
•
Products Storage
•
Pressure Vessels
•
Domestic and Industrial
Gas Equipment
•
McNAMAR
BOILER & TANK CO.
Box 868 -:- Tulsa, Okla.

kins, who has been manager of branch stores, will continue to make headquarters in Tulsa. Previous to his promotion Bastian was general salesman in East Texas, and he will continue to have headquarters at Longview.

M. L. Eubank, who has been a salesman in East Texas, has been appointed sales manager for the district and will have headquarters in Longview. W. O. Hardy, Tulsa salesman, has been named sales manager for the Kansas district, succeeding C. E. Robertson, and will have headquarters in Wichita.

Rockwell, Jr., Elected President Rockwell Manufacturing Company

W. F. Rockwell, Jr., formerly vice president and general manager of Rockwell Manufacturing Company, has been elected president of the company. W. F Rockwell, who has been president and board chairman, continues as chairman of the board. All other officers were re-elected at the recent annual meeting.

Houdry Names Louis Pineau Foreign Business Advisor

Louis Pineau has been named an advisor on foreign business to Houdry Process Corporation, and will maintain headquarters in Paris. He recently visited the Houdry laboratories, where his old-time acquaintance with Eugene J. Houdry and associates was renewed. Pineau sailed for Paris recently with Houdry.

Louis Pineau

Pineau is president of the Permanent Council of Petroleum Congresses (London), administrator general of Maison de la Chimie (Association of French Chemical Industries), and honorary president of the French Association of Petroleum Technologists. From 1920 to 1940, when he was removed by the Vichy regime, he directed the National Office of Liquid Fuels in the Ministry of Public Works. During the war he headed the joint Anglo-French Executive Committee for Oil from London.

Tube Turns Appoints McCutchan To Senior Engineering Post

Arthur McCutchan has been appointed senior research engineer of the product engineering and research department of Tube Turns (Inc.), Louisville, Kentucky.

Arthur McCutchan

McCutchan will be associated with A. R. C. Markl, chief research engineer, formerly mechanical engineer in charge of the pressure equipment and materials division of The M. W. Kellogg Company; Mason S. Noyes, research engineer, formerly senior marine engineer, Bureau of Ships, U. S. N., and Henry H. George, research engineer, formerly a physicist in the Jones and Laughlin research laboratory.

McCutchan attended the University of Michigan. He was associated for 20 years with the engineering division of Detroit Edison Company, specializing in the design of power-plant piping, before joining Tube Turns (Inc.). He is the author of numerous articles on piping flexibility, flange design, application of welding to high-pressure-high-temperature piping, and graphitization of high-temperature steam piping. He is co-author, with Sabin Crocker, of the chapter on Expansion and Flexibility in the Piping Handbook.

WANTED—MECHANICAL OR METALLURGICAL ENGINEER—familiar with the inspection of refinery fractionating equipment, furnaces, heat exchangers and pressure vessels and having experience to qualify for making recommendations for renewal or repairs to the equipment. Experience on catalytic cracking units preferred, but not essential. In replying, please send full details of experience and education. Snapshot (if available). Address: Box 10-R, c/o Petroleum Refiner, Houston, Texas.

WANTED:

Assistant to a head of a drafting department. Engineer, active, thorough type with ability to analyze facts and to express the results clearly in correspondence; to coordinate activities and to direct men; to follow procedures in orderly manner. Familiarity with drafting practice in the design of intricate piping systems and platework. Experience on gas plant, power plant or chemical plant design. Location: New York City. State qualifications and describe experience fully. Give age, salary expected. Address: Box 38-R, c/o Petroleum Refiner, Houston, Texas.

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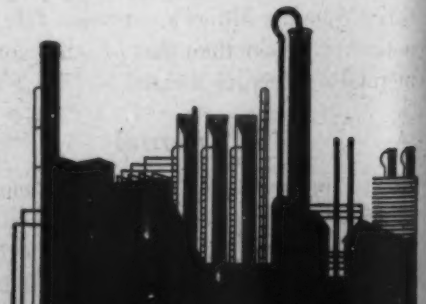

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